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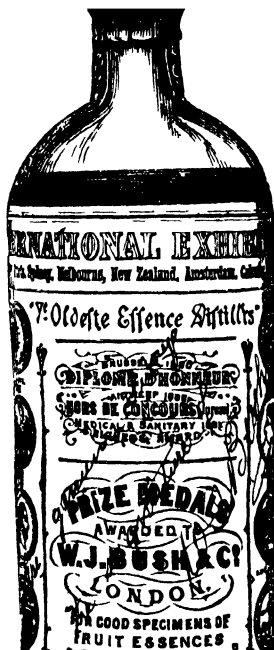
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ABSTRACTS OF PAPERS  
RELATING TO  
PHARMACY, MATERIA MEDICA, AND CHEMISTRY  
CONTRIBUTED TO BRITISH AND FOREIGN JOURNALS  
FROM JULY 1, 1914, TO JUNE 30, 1915,  
WITH THE  
TRANSACTIONS  
OF THE  
BRITISH PHARMACEUTICAL  
CONFERENCE

FIFTY SECOND ANNUAL MEETING

L O N D O N ,  
J U L Y 14, 1915.

IARI

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L O N D O N

J. &amp; A. CHURCHILL, 7, GREAT MARLBOROUGH STREET

1915.

# British Pharmaceutical Conference.

## CONSTITUTION.

Art I—This Association shall be called The British Pharmaceutical Conference, and its objects shall be the following—

- 1 To hold an annual Conference of those engaged in the practice, or interested in the advancement, of Pharmacy, with the view of promoting their friendly reunion, and increasing their facilities for the cultivation of Pharmaceutical Science
- 2 To determine what questions in Pharmaceutical Science require investigation, and when practicable, to allot them to individuals or committees to report thereon
- 3 To maintain uncompromisingly the principle of purity in Medicine
- 4 To form a bond of union amongst the various associations established for the advancement of the Science and Practice of Pharmacy, by receiving from them delegates to the annual Conference

Art. II—Membership in the Conference shall not be considered as conferring any guarantee of professional competency

## RULES.

1 Any person desiring to become a member of the Conference shall be nominated in writing by a member, and be balloted for at a general meeting of the members, two thirds of the votes given being needful for election. If the application be made during the recess, the Executive Committee may elect the candidate by a unanimous vote

2 The minimum subscription shall be 7s 6d annually, which shall be due in advance upon January 1.

3 Any member whose subscription shall be more than two years in arrear, after written application, shall be liable to be removed from the list by the Executive Committee. Members may be expelled for improper conduct by a majority of three fourths of those voting at a general meeting, provided that fourteen days' notice of such intention of expulsion has been sent by the Secretaries to each member of the Conference

4 Every association established for the advancement of Pharmacy shall, during its recognition by the Conference, be entitled to send delegates to the annual meeting

5 The Officers of the Conference shall be a President, a number of Vice presidents not exceeding six, by election the past Presidents (who shall be Vice presidents), a Treasurer, two General Secretaries, one Local Secretary, and nine other members, who shall collectively constitute the Executive Committee. Three members of the Executive Committee to retire annually by ballot, the remainder being eligible for re-election. They shall be elected at each annual meeting, by ballot of those present

6 At each Conference it shall be determined at what place and time to hold that of the next year

7 Two members shall be elected by the Conference to audit the Treasurer's accounts, such audited accounts to be presented annually

8. The Executive Committee shall present a report of proceedings annually

9 These rules shall not be altered except at an annual meeting of the members

10 Reports on subjects entrusted to individuals or committees for investigation shall be presented to a future meeting of the Conference, whose property they shall become. All reports shall be presented to the Executive Committee at least fourteen days before the annual meeting

\* \* \* Authors are specially requested to send the titles of their Papers to The Hon. Gen. Secy Brit Pharm Conf, 17, Bloomsbury Square, London, W C, two or three weeks before the Annual Meeting. The subjects will then be extensively advertised, and thus full interest will be secured

## NOMINATION FORM.

*I desire to nominate*

*Name)*

*Address)*

*as a Member of the British Pharmaceutical Conference.*

*Member*

*Date*

This or any similar form may be filled up, and forwarded to The Hon. Gen. Secy, Brit Pharm Conf, 17, Bloomsbury Square, London, W C

Pupils and Assistants, as well as Principals, are invited to become members.

# BRITISH PHARMACEUTICAL CONFERENCE

## OFFICERS FOR 1915-1916.

### PRESIDENT.

MAJOR E. SAVILLE PECK, Cambridge.

### ACTING PRESIDENT.

JOHN C. UMNEY, F.C.S., London.

### VICE-PRESIDENTS,

*Who have filled the office of President.*

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E. M. HOLMES, F.L.S., Sevenoaks.  
G. CLARIDGE DRUCE, M.A., J.P., Oxford.  
T. H. W. IDRIS, F.C.S., London.  
W. A. H. NAYLOR, F.I.C., London.  
THOS. TYRER, F.I.C., London.  
ROBERT WRIGHT, F.C.S., Buxton.  
J. F. TOCHER, D.Sc., F.I.C., Aberdeen.  
FRANCIS RANSOM, F.C.S., Hitchin.  
W. F. WELLS, Dublin.  
SIR EDWARD EVANS, J.P., Liverpool.  
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### VICE-PRESIDENTS.

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D. M. WATSON, Dublin.  
EDMUND WHITE, B.Sc., F.I.C., London.  
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### HONORARY TREASURER.

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### HONORARY GENERAL SECRETARIES.

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### HONORARY LOCAL SECRETARY.

H. HUMPHREYS JONES, Liverpool.

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T. O. BARLOW, Southsea.	D. HOOPER, F.I.C., Weston-super-
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F. W. GAMBLE, London.	W. F. J. SHEPHEARD, Chester.
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### AUDITORS.

I. BOURDAS, London, and W. F. GULLIVER, London.

### HONORARY SECRETARIES FOR OVERSEAS DOMINIONS.

For BENGAL . . . . .	W. T. GRICE, Calcutta.
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„ NEW ZEALAND . . . . .	R. WILKINSON, Dunedin.
„ VICTORIA . . . . .	C. L. BUTCHERS, Melbourne.

# BRITISH PHARMACEUTICAL CONFERENCE.

INAUGURAL MEETING HELD AT NEWCASTLE-ON-TYNE IN 1863.

<i>Years</i>	<i>Places of Meeting.</i>	<i>Presidents.</i>	<i>Vice-Presidents.</i>	<i>Local Secretaries.</i>
1864	Bath . . .	HENRY DEANE, F.L.S.	Prof. BENTLEY, F.L.S. Dr. EDWARDS, F.C.S. R. W. GILES, F.C.S.	J. C. POOLEY.
1865	Birmingham	HENRY DEANE, F.L.S.	Prof. REDWOOD, F.C.S. Prof. BENTLEY, F.L.S. Dr. EDWARDS, F.C.S. W. SOUTHALL. J. P. TYLER.	W. SOUTHALL, Jun.
1866	Nottingham	Prof. BENTLEY, F.L.S.	Dr. EDWARDS, F.C.S. D. HANBURY, F.R.S. SAMUEL PARR.	J. H. ATHERTON, F.C.S.
1867	Dundee . .	Prof. BENTLEY, F.L.S.	W. W. STODDART, F.G.S. D. HANBURY, F.R.S. J. INCE, F.L.S. D. RUSSELL.	J. HODGE.
1868	Norwich .	DANIEL HANBURY, F.R.S.	W. W. STODDART, F.G.S. R. FITCH, F.G.S. J. INCE, F.L.S. W. W. STODDART, F.G.S.	F. SUTTON, F.C.S.
1869	Exeter . .	DANIEL HANBURY, F.R.S.	J. R. YOUNG. G. COOPER. H. S. EVANS, F.C.S. J. INCE, F.L.S.	M. HUSBAND.
1870	Liverpool .	W. W. STODDART, F.C.S.	W. W. STODDART, F.G.S. J. ABRAHAM. H. C. BAILDON. H. S. EVANS, F.C.S.	E. DAVIES, F.C.S. J. DUTTON (Birkenhead).
1871	Edinburgh .	W. W. STODDART, F.C.S.	J. INCE, F.L.S. J. ABRAHAM. H. C. BAILDON. J. INCE, F.L.S.	J. MACKAY, F.C.S.
1872	Brighton .	H. B. BRADY, F.R.S.	J. WILLIAMS, F.C.S. J. INCE, F.L.S. R. REYNOLDS, F.C.S. W. D. SAVAGE.	T. GLAISYER.
1873	Bradford .	H. B. BRADY, F.R.S.	J. WILLIAMS, F.C.S. T. H. HILLS, F.C.S. R. REYNOLDS, F.C.S. F. M. RIMMINGTON, F.C.S.	R. PARKINSON, Ph.D.
1874	London . .	THOS. B. GROVES, F.C.S.	J. WILLIAMS, F.C.S. T. H. HILLS, F.C.S. R. REYNOLDS, F.C.S. CHAS. H. SAVORY.	M. CARTEIGHE, F.C.S.
1875	Bristol . .	THOS. B. GROVES, F.C.S.	J. WILLIAMS, F.C.S. T. H. HILLS, F.C.S. R. REYNOLDS, F.C.S. CHAS. BOORNE.	J. PITMAN.
1876	Glasgow .	Prof. REDWOOD, F.C.S.	PETER SQUIRE, F.L.S. T. H. HILLS, F.C.S. R. REYNOLDS, F.C.S. E. C. C. STANFORD, F.C.S. D. FRAZER.	A. KINNINMONT.
1877	Plymouth .	Prof. REDWOOD, F.C.S.	T. H. HILLS, F.C.S. R. REYNOLDS, F.C.S. A. P. BALKWILL.	R. J. CLARK
1878	Dublin . .	G. F. SCHACHT, F.C.S.	J. WILLIAMS, F.C.S. Prof. TICHBORNE, F.C.S. R. REYNOLDS, F.C.S. R. W. PRING, L.A.H.D.	W. HAYES.
1879	Sheffield .	G. F. SCHACHT, F.C.S.	J. WILLIAMS, F.C.S. Prof. TICHBORNE, F.C.S. R. REYNOLDS, F.C.S. W. WARD, F.C.S.	H. MALEHAM.
1880	Swansea .	W. SOUTHALL, F.L.S.	J. WILLIAMS, F.C.S. R. REYNOLDS, F.C.S. G. W. SANDFORD. W. WARD, F.C.S.	J. HUGHES.
1881	York . .	R. REYNOLDS, F.C.S.	N. M. GROSE. Prof. ATTFIELD, F.R.S. R. DAVISON. N. M. GROSE.	J. SOWEAY.
1882	Southampton	Prof. ATTFIELD, F.R.S.	C. UMNEY, F.C.S. R. CHIPPERFIELD. T. GREENISH, F.C.S. Prof. TICHBORNE, LL.D. J. R. YOUNG.	O. R. DAWSON.

# BRITISH PHARMACEUTICAL CONFERENCE.

<i>Years.</i>	<i>Places of Meeting.</i>	<i>Presidents.</i>	<i>Vice-Presidents.</i>	<i>Local Secretaries.</i>
1883	Southport .	Prof. ATTFIELD, F.R.S.	M. CARTEIGHE, F.C.S. W. V. RADLEY. C. UMNEY, F.C.S. J. R. YOUNG.	WM. ASHTON.
1884	Hastings .	J. WILLIAMS, F.C.S.	S. R. ATKINS. J. BELL. M. CARTEIGHE, F.C.S. J. R. YOUNG.	F. ROSSITER.
1885	Aberdeen .	J. B. STEPHENSON.	F. B. BENDER, F.C.S. M. CARTEIGHE, F.C.S. C. EKIN, F.C.S. J. P. KAY.	A. STRACHAN.
1886	Birmingham	T. GREENISH, F.C.S.	T. BARCLAY. F. B. BENDER, F.C.S. M. CARTEIGHE, F.C.S. C. EKIN, F.C.S.	CHAS. THOMPSON.
1887	Manchester	S. R. ATKINS, J.P.	M. CARTEIGHE, F.C.S. S. PLOWMAN, F.R.C.S. C. SYMES, Ph.D. G. S. WOOLLEY.	F. B. BENDER, F.C.S.
1888	Bath . .	F. B. BENDER, F.C.S.	M. CARTEIGHE, F.C.S. S. PLOWMAN, F.R.C.S. C. SYMES, Ph.D. W. MARTINDALE, F.C.S.	H. HUTTON.
1889	Newcastle-on-Tyne	C. UMNEY, F.I.C., F.C.S.	M. CARTEIGHE, F.C.S. S. PLOWMAN, F.R.C.S. C. SYMES, Ph.D. N. H. MARTIN, F.L.S.	T. M. CLAGUE.
1890	Leeds . .	C. UMNEY, F.I.C., F.C.S.	M. CARTEIGHE, F.C.S. S. PLOWMAN, F.R.C.S. A. KINNINMONT, F.C.S. W. SMEETON.	F. W. BRANSON, F.C.S.
1891	Cardiff . .	W. MARTINDALE, F.C.S.	M. CARTEIGHE, F.C.S. A. KINNINMONT, F.C.S. J. C. THRESH, M.B., D.Sc. J. MUNDAY.	ALFRED COLEMAN.
1892	Edinburgh .	E. C. C. STANFORD, F.C.S.	M. CARTEIGHE, F.C.S. W. GILMOUR, F.R.S.E. J. C. THRESH, M.B., D.Sc. J. R. YOUNG, J.P.	PETER BOA.
1893	Nottingham	OCTAVIUS CORDER.	M. CARTEIGHE, F.C.S. J. LAIDLAW EWING. W. HAYES. R. FITZ HUGH.	C. A. BOLTON.
1894	Oxford . .	N. H. MARTIN, F.L.S., F.R.M.S.	M. CARTEIGHE, F.C.S. R. H. DAVIES, F.C.S. W. HAYES. G. T. PRIOR.	H. MATHIJS.
1895	Bournemouth	N. H. MARTIN, F.L.S., F.R.M.S.	M. CARTEIGHE, F.C.S. J. LAIDLAW EWING. W. HAYES. J. A. TOONE.	STEWART HARDWICK.
1896	Liverpool .	W. MARTINDALE, F.C.S.	M. CARTEIGHE, F.C.S. J. LAIDLAW EWING. M. CONROY, F.C.S. W. HAYES.	T. H. WARDLE-WORTH.
1897	Glasgow .	Dr. C. SYMES.	WALTER HILLS. J. LAIDLAW EWING. W. F. WELLS. R. MCADAM.	H. O. DUTTON. (Birkenhead). J. A. RUSSELL.
1898	Belfast . .	Dr. C. SYMES.	WALTER HILLS. J. LAIDLAW EWING. J. C. C. PAYNE, J.P. W. F. WELLS.	R. W. MCKNIGHT W. J. RANKIN.
1899	Plymouth .	J. C. C. PAYNE, J.P.	WALTER HILLS, F.C.S. R. J. DOWNES. JOHN MOSS, F.I.C., F.C.S. C. J. PARK.	J. DAVY TURNER.
1900	London . .	E. M. HOLMES, F.L.S.	R. J. DOWNES. WALTER HILLS, F.C.S. JOHN MOSS, F.I.C., F.C.S. J. F. HARRINGTON.	W. WARREN. HERBERT CRACKNELL.
1901	Dublin . .	G. C. DRUCE, M.A., F.L.S.	G. T. W. NEWSHOLME, F.C.S. G. D. BEGGS, M.P.S.I. PETER BOA, F.C.S. Prof. TICHBORNE, Ph.D.	J. I. BERNARD.



# BRITISH PHARMACEUTICAL CONFERENCE.

<i>Years.</i>	<i>Places of Meeting.</i>	<i>Presidents.</i>	<i>Vice-Presidents.</i>	<i>Local Secretaries.</i>
1902	Dundee .	G. C. DRUCE, M.A., F.L.S.	G. T. W. NEWSHOLME.  G. D. BEGGS, M.P.S.I. CHAS. KERR. W. A. H. NAYLOR, F.I.C.	W. CUMMINGS.
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1904	Sheffield .	T. H. W. IDRIS, M.P., F.C.S.	G. D. BEGGS, M.P.S.I. D. B. DOTT, F.R.S.E., W. A. H. NAYLOR, F.I.C. G. T. W. NEWSHOLME, F.C.S.	H. ANTCLIFFE.
1905	Brighton .	W. A. H. NAYLOR, F.I.C., F.C.S.	F. RANSOM, F.C.S. R. A. ROBINSON, J.P. D. B. DOTT. J. MONTGOMERY. W. H. GIBSON. F. RANSOM, F.C.S. H. G. GREENISH.	W. W. SAVAGE. C. G. YATES.
1906	Birmingham	W. A. H. NAYLOR, F.I.C., F.C.S.	R. A. ROBINSON, J.P. D. B. DOTT. W. F. WELLS. F. RANSOM. H. G. GREENISH, F.I.C. T. BARCLAY.	C. THOMPSON.
1907	Manchester .	THOS. TYRER, F.I.C., F.C.S.	R. A. ROBINSON, J.P. D. B. DOTT. J. A. WALSH. F. RANSOM. H. G. GREENISH, F.I.C. G. S. WOOLLEY.	W. KIRKBY.
1908	Aberdeen .	ROBT. WRIGHT, F.C.S.	J. R. YOUNG. G. LUNAN. J. A. WALSH. W. GILES. H. G. GREENISH, F.I.C. F. RANSOM.	W. F. HAY.
1909	Newcastle .	J. F. TOOHER, B.Sc., F.I.C.	J. R. YOUNG. G. LUNAN. J. SMITH. GEORGE WEDDELL. F. RANSOM. H. G. GREENISH, F.I.C.	T. M. CLAGUE. H. W. NOBLE.
1910	Cambridge .	FRANCIS RANSOM, F.C.S.	J. F. HARRINGTON. J. P. GILMOUR. J. REYNOLDS GREEN, Sc.D., F.R.S. JOHN SMITH. EDMUND WHITE, B.Sc., F.I.C.	A. A. DECK. T. J. MALLETT.
1911	Portsmouth	W. F. WELLS.	H. G. GREENISH, F.I.C. J. F. HARRINGTON. J. P. GILMOUR. H. G. GREENISH, F.I.C. JOHN SMITH. EDMUND WHITE, B.Sc., F.I.C.	T. O. BARLOW. T. POSTLE- THWAIT
1912	Edinburgh .	SIR EDWARD EVANS, J.P.	T. A. WHITE. C. B. ALLEN. SIR WILLIAM BAXTER. J. LAIDLAW EWING. J. P. GILMOUR. H. G. GREENISH, F.I.C. EDMUND WHITE, B.Sc., F.I.C.	THOS. STEPHEN- SON.
1913	London .	JOHN C. UMNEY, F.C.S.	C. B. ALLEN. SIR WILLIAM BAXTER, J.P. J. P. GILMOUR. H. G. GREENISH, F.I.C. E. SAVILLE PECK, M.A. EDMUND WHITE, B.Sc., F.I.C.	W. J. UGLOW WOOLCOCK

<i>Years.</i>	<i>Places of Meeting.</i>	<i>Presidents.</i>	<i>Vice-Presidents.</i>	<i>Local Secretaries.</i>
1914	Chester . .	E. H. FARR, F.C.S.	Sir WILLIAM BAXTER, J.P. J. P. GILMOUR E. F. HARRISON, B.Sc., F.I.C. E. SAVILLE PECK, M.A. W. F. J. SHEPHEARD EDMUND WHITE, B.Sc., F.I.C.	R. 'CECIL OWEN, B.Sc.
1915	London . .	Major E. SAVILLE PECK.	J. P. GILMOUR. E. F. HARRISON, B.Sc. F.I.C. F. B. POWER, Ph.D. D. M. WATSON. EDMUND WHITE, B.Sc., F.I.C. G. WHITFIELD	- -

#### TREASURERS (One)

1863 to 1870, H. B. BRADY, F.R.S.	1886 to 1893, R. H. DAVIES, F.I.C., F.C.S.
1870 to 1877, GEORGE F. SCHACHT, F.C.S.	1893 to 1898, JOHN MOSS, F.I.C., F.C.S.
1877 to 1884, C. ERIN, F.C.S.	1898 to 1912, JOHN C. UMNEY, F.C.S.
1884 to 1888, C. UMNEY, F.I.C., F.C.S.	1912 to D. LLOYD HOWARD, F.C.S.
1888 to 1890, W. MARTINDALE, F.C.S.	

#### HONORARY GENERAL SECRETARIES (Two).

1863 to 1880, Prof. ATTFIELD, Ph.D., F.R.S.	1886 to 1901, W. A. H. NAYLOR, F.I.C., F.C.S.
1863 to 1871, RICHARD REYNOLDS, F.C.S.	1890 to 1903, F. RANSOM, F.C.S.
1871 to 1884, F. BADEN BENGER, F.C.S.	1903 to 1909, EDMUND WHITE, B.Sc., F.I.C.
1880 to 1882, M. CARTERIGHE, F.C.S.	1901 to 1912, E. SAVILLE PECK, M.A.
1882 to 1886, SIDNEY FLOWMAN, F.R.C.S.	1909 to HORACE FINCHMORE B.Sc., F.I.C.
1884 to 1890, JOHN C. TURESH, M.B., D.Sc.	1912 to R. R. BENNETT, B.Sc., F.I.C.

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The duties the Local Corresponding Secretaries have undertaken to discharge are briefly as follows:—

(a) To bring under the notice of pharmacists, principals, and their assistants, in their districts, who are unassociated with the Conference, the advantage of membership with it, and by personal effort to try and induce them to join.

(b) To assist in stimulating research by asking pharmacists, who have the time, ability, and disposition, to contribute from time to time a paper or useful note to the annual meetings.

(c) To endeavour to induce defaulters to continue their membership.

(d) To take generally a watchful and sympathetic interest in the affairs of the Conference. To render those services voluntarily at times convenient to themselves and as opportunity offers.

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AN ORGANIZATION ESTABLISHED IN 1863 FOR THE ENCOURAGEMENT OF PHARMACEUTICAL RESEARCH, AND THE PROMOTION OF FRIENDLY INTERCOURSE AND UNION AMONGST PHARMACISTS.

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THE most important ways in which a member can aid the objects of the Conference are by introducing new members, suggesting subjects for investigation, working upon subjects suggested by himself or by others, contributing information tending to throw light on questions relating to adulterations and impurities, or collecting and forwarding specimens whose examination would afford similar information. Personal attendance at the yearly gatherings, or the mere payment of the annual subscription, will also greatly strengthen the hands of the executive.

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The annual meeting for 1916 will be held at Liverpool.

Ladies and gentlemen desiring to join the Conference can be nominated at any time on applying to the Secretaries, or any other officer or member. The yearly subscription is payable in advance, on January 1st. The amount, which includes free delivery of the Year-Book, is fixed at a minimum of 7s. 6d. for members residing within the Postal Union. Further information may be obtained from

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Members of the Conference receive annually *The Year-Book of Pharmacy*: a volume of about 600 pages, containing the Proceedings at the yearly meeting, and abstracts of scientific papers published, throughout the world. These include an account of current work on Chemistry, Essential Oils, Materia Medica, Galenical Pharmacy, Dispensing Notes, New Remedies, New Apparatus, New Formulæ and New Processes. The necessary fund for accomplishing this object consists solely of the subscriptions of members. The Executive Committee, therefore, call on every pharmacist—principal, assistant, or pupil—to offer his name for election, and on every member to make an effort to obtain more members. The price of the Year-Book to non-members is ten shillings. The constitution and rules of the Conference, and a convenient form of nomination, will be found at page ii.

# YEAR-BOOK OF PHARMACY

## CHEMISTRY

### ALKALOIDS

**Aconitine, Oxidation of.** G. Barger and Ellen Field. (*Trans. Chem. Soc.*, 1915, **107**, 231.) When aconitine is oxidized in acetone solution with  $\text{KMnO}_4$  in presence of glacial  $\text{HC}_2\text{H}_3\text{O}_2$ , 90 per cent. of the theoretical yield of oxonitin is obtained. Oxonitin crystallizes best from boiling glacial  $\text{HC}_2\text{H}_3\text{O}_2$  after addition of acetone, forming thick prisms, m.p.  $276^\circ$ – $277^\circ\text{C}$ . When heated with HI and P, it is converted into a substance, crystallizing from alcohol in prisms, m.p.  $121^\circ$ – $122^\circ\text{C}$ , b.p. about  $200^\circ\text{C}$ . at 15 mm. The mean results of a number of analyses of recrystallized oxonitin gave C, 60.51, H, 6.66,  $(\text{CH}_3\text{O})$ , 18.5 per cent.

**Alkaloidal Assays, A Possible Source of Error in.** P. A. W. Self. (*Pharm. J.*, 1915 [4], **40**, 585.) That certain of the alkaloids have the power of expelling  $\text{NH}_3$  from its salts is well known, but it does not appear to be equally well known that probably this power is possessed by nearly all alkaloids. Although ammonia is a much stronger base than most fixed alkaloids, its great volatility largely counterbalances this fact, for it is a matter of common knowledge that a feeble base or acid is capable of expelling from combination a much stronger member of its class, if the former is fixed and the latter volatile at the temperature employed.

In an alkaloidal assay, if the alkaloid is liberated by ammonia and the volatile solvent employed in the final shaking-out is not washed with water, but is run directly into a dish and evaporated, there is considerable danger of a small volume of the

aqueous layer (containing an ammonium salt) being carried into the dish with the alkaloid. The natural result is that, as pointed out above, a certain amount of interaction takes place during evaporation of the solvent, with consequent loss of ammonia and formation of an alkaloidal salt. The residue finally obtained, therefore, consists of a mixture of free alkaloid and alkaloidal salt, and the titration, of course, only gives the amount of the former.

It is interesting to note that here the result obtained is too low, whereas when the alkaloid is not titrated, but weighed, the introduction of ammonium salt causes the result to become too high. The error in the case of titration may, however, be much more serious than when the alkaloid is weighed, since, owing to the high molecular weights of all alkaloids, a very small quantity of a salt of ammonia is capable of causing the neutralization of a comparatively large weight of free alkaloid if interaction is complete. Thus one part of AmCl will interact with 12 parts of aconitine, 5.4 parts of atropine, or 6.3 parts of strychnine.

A number of quantitative experiments are given bearing out these facts.

In the first series of experiments, weighed amounts of pure alkaloids were dissolved in a little  $\text{CHCl}_3$  or  $\text{Et}_2\text{O}$  a small quantity of a weak solution of AmCl added, and the whole evaporated to dryness on a water-bath and titrated in the usual way. In each case a similar experiment was performed, in which the AmCl was omitted, in order to eliminate any other error and check the purity of the alkaloid.

It was found that the interaction was greatest in the case of atropine, the reaction being practically complete in both experiments. With aconitine the larger amount of apparent loss in the second experiment was probably due to the use of  $\text{Et}_2\text{O}$  causing better contact between the reacting substances than when  $\text{CHCl}_3$  was employed, a result which might very well be expected. Strychnine gave least interaction, this no doubt being due to its extreme insolubility in water rendering contact with the AmCl very difficult. In all the experiments, however, considering the minute quantities of AmCl used, the apparent loss of alkaloid was comparatively large.

Assays of belladonna leaves were next carried out, by the method of the B.P. 1914, but in some experiments washing the chloroform used in the final extraction with water, and in others allowing small quantities of the aqueous layer to pass into the

TABLE I

Alkaloid used.	Details of Experiment.	Amount Taken	Amount found by Titration.	Apparent Loss.
Atropine .	Alkaloid dissolved in 5 c.c. of ether, and about 4 milligrammes of ammonium chloride in 1 c.c. of water added	0.0489 Gm.	0.0243 Gm.	0.0246 Gm.
Atropine .	Alkaloid dissolved in 5 c.c. of chloroform, and about 2 milligrammes of ammonium chloride in 0.5 c.c. of water added	0.0451 Gm.	0.0333 Gm.	0.0118 Gm.
Aconitine	Alkaloid dissolved in 3 c.c. of chloroform and about 4 milligrammes of ammonium chloride in 1 c.c. of water added	0.0925 Gm.	0.0839 Gm.	0.0086 Gm.
Aconitine	Alkaloid dissolved in 10 c.c. of ether and about 2 milligrammes of ammonium chloride in 0.5 c.c. of water added	0.0502 Gm.	0.0336 Gm.	0.0166 Gm.
Strychnine	Alkaloid mixed with 3 c.c. of chloroform and about 8 milligrammes of ammonium chloride in 2 c.c. of water added	0.0956 Gm.	0.0809 Gm.	0.0147 Gm.
Strychnine	Alkaloid mixed with 10 c.c. of ether and about 2 milligrammes of ammonium chloride in 0.5 c.c. of water added	0.0479 Gm.	0.0451 Gm.	0.0028 Gm.

dish with the alkaloids. The method adopted for the purpose of eliminating any possibility of variation in the first stages of the assay, in comparative experiments, was as follows:—Fifty grammes of the powdered leaf was taken, the extraction with  $\text{Et}_2\text{O}-\text{CHCl}_3$  and shaking-out with acid performed with five times the Pharmacopœial quantities of all the reagents, and the acid extract then made up to 250 c.c. ; 50 c.c. of this liquid were taken for the final stage of each separate experiment, and the exact volumes of  $\text{CHCl}_3$  prescribed by the B.P. used in shaking-out. Three samples of leaf were employed, and the circumstances were varied, as described in Table II.

In experiments (3) and (4) the separation of the  $\text{CHCl}_3$  was done with great care, the only aqueous liquid carried into the

dish being that unavoidably removed from the sides of the separator by friction; the results are, however, appreciably low. In experiments (6), (7), and (9) small quantities of the aqueous layer (containing, of course, some  $\text{Am}_2\text{SO}_4$ ) were added intentionally, the other circumstances being the same as in experiments (3) and (4), and in all three cases the errors produced were very serious. It is worthy of note that in this assay the fact that  $\text{Et}_2\text{O}$  is added to the alkaloidal residue and then evaporated off again appears to increase any error due to the presence of ammonium salt by promoting better contact, since the apparent loss in experiment (9), where no  $\text{Et}_2\text{O}$  was used, was comparatively much smaller than in experiment (6). It is therefore quite evident, from the figures obtained in both sets of experiments, that in any alkaloidal assay in which the alkaloidal residue is titrated great care must be exercised to avoid the introduction into the latter of even as little as 1 Mgm. of an ammonium salt. It is therefore extremely advisable to wash with water the solution of alkaloid in volatile solvent.

TABLE II

Number of Sample.	Number of Experiment.	Circumstances of Experiment.	Percentage of Alkaloid Found.	Error.
I.	1	Chloroformic solution of alkaloid washed with water . . . .	0.295	—
	2	Chloroformic solution of alkaloid washed with water . . . .	0.289	—
	3	Chloroform run very carefully into dish without washing . . . .	0.278	0.014
	4	Chloroform run very carefully into dish without washing . . . .	0.278	0.014
II.	5	Chloroformic solution of alkaloid washed with water . . . .	0.402	
	6	As expts. (3) and (4), but about 0.3 c.c. of aqueous layer run into dish in addition . . . .	0.197	0.205
	7	As expt. (6), but 0.5 c.c. of aqueous layer added . . . .	0.191	0.211
III.	8	Chloroformic solution of alkaloid washed with water . . . .	0.278	—
	9	As expt. (6), but 0.25 c.c. of aqueous layer added, and alkaloid not treated with ether finally .	0.208	0.070

From this point of view it is somewhat unfortunate that in the B.P. assay processes for belladonna leaf and tincture and

dry extract of belladonna no directions are given for washing the  $\text{CHCl}_3$  solution of alkaloid before evaporation and titration, while it is certainly very difficult to understand why the precaution which was omitted in these cases should have been taken in the assay of liquid extract of belladonna. With regard to aconite and its preparations, while the prescribed filtration of the  $\text{Et}_2\text{O}$ , if properly carried out, appears to render any appreciable error unlikely, yet great care must be taken that none of the aqueous layer passes through the filter, otherwise, owing to the high molecular weight of aconitine, a very serious error may result.

**Alkaloids, Function of, in *Papaver Somniferum*.** A. Mueller. (*Archiv. Pharm.*, 1914, **252**, 280.) The seeds of *P. somniferum* contain no alkaloids, and none appear in the plant until 14 days after germination. The amount of alkaloid increases with the growth of the plant, until after flowering, when the concentration of albumin in the seeds commences. As this progresses during the ripening of the seeds, the amount of alkaloids diminishes in the plant. It seems that the plant bases are not excretory products; but serve as sources of N for the formation of albuminoids in the seeds.

**Alkaloids, Modification of the Silico-tungstate Method for Determining.** -- Ferencz and -- David. (*Pharm. Post, Schwiz. Apoth. Zeit.*, 1914, **52**, 686.) The precipitate of the alkaloidal silico-tungstate obtained in the usual manner is collected on a filter and transferred, after draining, to a flask. It is then evenly suspended in 5 c.c. of water and treated with 10 c.c. of 1:10 NaOH solution, and shaken for 10 minutes; 15 Gm. of NaCl is then added. In the case of atropine and brucine, 100 c.c. of  $\text{Et}_2\text{O}$  is then added, or with nicotine, 50 c.c. of  $\text{Et}_2\text{O}$  and 50 c.c. of petroleum ether. The whole is then well shaken together for 10 minutes, and set aside until the immiscible solvent separates clear. Exactly 50 c.c. is then pipetted off, and treated with excess of N/100 HCl and titrated back with N/100 NaOH, with iodeosin indicator in the usual manner. The results obtained with pure nicotine, brucine and atropine are satisfactory. The method is also available for the determination of these [and similar] alkaloids in extracts. This modification is much more rapid than other methods with silico-tungstic acid. (See also *Y.B.*, 1913, 3.)



**Alkaloids, Colloidal State of : Relations between Surface Tension, Size of Particles and Toxicity.** I. Traube and N. Onodera. (*Inter. Zeit. Phys. Chem. Biol. : J.S.C.I.* 1915, **34**, 509.) Alkaloids of high molecular weight, such as atropine and quinine, are present in solution in a colloidal state ; the corresponding salts form true solutions. The surface tension of water is scarcely affected when alkaloidal salts are dissolved in it, but is diminished on adding subsequently a small quantity of alkali, owing to liberation of free alkaloid which assumes a colloidal condition ; at the same time the toxicity of the solution increases. Many free alkaloids are unstable in solution, the small particles aggregating in a few hours into large masses, with consequent increase of surface tension and decrease of toxicity. On adding a little alkali to such an attenuated alkaloid solution the aggregates disperse, the surface tension diminishes, and the toxicity reaches or even surpasses its former intensity. The localization of action of the various alkaloids may be partly due to the variation in the alkalinity of the different organs of the body. The toxicity of solutions of some alkaloids, including quinine, is increased by boiling and subsequently cooling, with corresponding alterations in the size of the colloidal particles and the surface tension. The antagonistic action of pilocarpine on atropine is accompanied by similar changes. With all alkaloids the alteration in surface tension is the chief factor in determining the toxicity. It is suggested that any alteration in the surface tension disturbs the equilibrium or affects the normal velocity of the reactions taking place in the organism, this becoming apparent as a toxic effect, the toxicity of alkaloids being thus due chiefly to the physical instead of to the chemical changes produced. When an electric current is passed through a solution of an alkaloid, the smaller particles wander towards the cathode, whilst the larger aggregates accumulate at the anode.

**Alkaloids, Catalytic Action of, on Precipitation, Oxidation and Saponification Processes.** I. Traube and N. Onodera. (*Inter. Zeits. Phys. Chem. Biol.*, 1914, **1**, 148 ; *J.S.C.I.*, 1915, **34**, 550). Alkaloids, although univalent, greatly accelerate the precipitation of  $\text{As}_2\text{S}_3$  from its colloidal solution and also the oxidation of  $\text{H}_2\text{C}_2\text{O}_4$  by  $\text{KMnO}_4$ . A few alkaloids inhibit the saponification of ethyl acetate by  $\text{KOH}$ , whilst the majority accelerate it. Pilocarpine has a strong accelerating action, whilst atropine has an inhibitory action on the saponification

process, though both alkaloids act similarly with respect to precipitation and oxidation processes. It is suggested that the antagonistic physiological action of these two alkaloids is possibly correlated with their action on hydrolytic processes.

**Alkaloids, Action of, on Germinating Seeds.** W. Sigmund. (*Biochem. Zeit.*, **62**, 299, *Chem. Abst.*, 1914, **8**, 2,560.) Conine is comparatively slightly injurious for the germination of seeds. Nicotine, in solutions containing 0.004 mol. of the HCl salt per litre, has the same action as conine-HCl in equivalent amounts. In stronger solutions (0.01, 0.02 and 0.04 mol. per litre) it is much more poisonous than an equivalent solution of conine-HCl. Piperine and piperinic acid are, as far as they are soluble in cold  $H_2O$ , up to 0.004 mol. per litre, more injurious; piperidine, in concentration of 0.001 mol. per litre, is relatively less poisonous. The simultaneous action of the decomposition products of piperine, piperidine and piperinic acid, in equimolecular proportions, increases the injurious effect of both. Atropine in less than 0.01 mol. sulphate per litre is little poisonous; between 0.02 and 0.04 mol. it has an extensive harmful action. Of the decomposition products, tropine is less, tropic acid more toxic than atropine itself; the most toxic product is atropic acid. Hyoscyamine has practically the same effect as atropine. Pilocarpine is relatively slightly poisonous; small amounts of atropine do not neutralize the action of large quantities of pilocarpine, as is the case in animals. Cocaine is slightly poisonous below 0.02 mol. per litre; 0.04 mol. HCl salt has a noticeable effect. It is more toxic than benzoylecgonine or ecgonine. Lupinine, lupinidine and the isomeric sparteine are not injurious in solutions up to 0.01 mol. per litre. Cytisine in proportion of 0.02 mols. per litre  $H_2O$  of HCl salts is not very injurious. Cinchonine, cinchonidine and quinine are strongly toxic in solutions of 0.02 to 0.04 mol. sulphate; quinine is the most poisonous of the three. Morphine and narcotine in solutions of 0.04 mol. HCl salt per litre  $H_2O$  are injurious, morphine being the more harmful. Berberine-HCl is a strong poison, acting in a solution of 0.0045 mol. per litre  $H_2O$ . Strychnine nitrate is active in 0.02 to 0.04 mol. per litre. Brucine-HCl is less poisonous than strychnine. Aconitine, even in concentration of 0.02 mols. HCl salt per litre  $H_2O$ , is little injurious. Veratrine is a little more active. Solanine and solanidine are very slightly harmful to germination.

**Apomorphine, Action of Acetic Anhydride on.** M. Tiffeneau and Porcher. (*Bull. Soc. Chim.*, 1915, 17, 114–119.) The apomorphine was acetylated by heating the base or hydrochloride with its acetic anhydride on a water-bath for 20–30 hours. The product consisted mainly of a mixture of about equal parts of diacetyl- and triacetyl-apomorphine. The fission of the nitrogen ring in the latter substance had deprived it of basic properties, and it was separated from the basic diacetyl-derivative by taking advantage of this fact. Diacetyl-apomorphine crystallized from a mixture of acetic ether and petroleum in needles, m.p. 129°C. Its salts possess the same physiological action as those of apomorphine and have the advantage of being stable in solution. With methyl iodide, diacetyl-apomorphine gives a methiodide, m.p. 233°C. Triacetyl-apomorphine has the m.p. 137°C., is optically inactive, insoluble in water and acids, and does not possess the emetic properties of the diacetyl-derivative. A crystalline apomorphine in hexagonal scales, m.p. 195°C., free from solvent of crystallization, has been obtained by crystallizing from a mixture of  $\text{CHCl}_3$  and petroleum ether.

**Apomorphine Hydrochloride, Sensitive Reaction for.** L. Grimbert and A. Leclerc. (*J. Pharm. Chim.*, 1915, 11, 23.) To 5 c.c. of a dilute solution of apomorphine hydrochloride 5 drops of saturated aqueous solution of  $\text{HgCl}_2$  and 5 drops of  $\text{NaC}_2\text{H}_3\text{O}_2$  are added, and the mixture is boiled for a few seconds. When cold, 2 c.c. of amylic alcohol is added. In the presence of 1:500,000 of apomorphine a blue colour will be evident in the latter solvent. Mayer's solution gives no reaction with this dilution. A confirmatory but not specific and less sensitive reaction is the reduction of ammoniacal  $\text{AgNO}_3$  by apomorphine.

**Aporeine and Its Salts, from Papaver dubium.** V. Pavesi. (*Gazz. Chim. Ital.*, 44, I, 398–405; 37, 1; *Chem. Abstr.* 1914, 8, 3017.) Extraction of the alkaloid after treating with spent lime and drying in the air with  $\text{Et}_2\text{O}$  or  $\text{EtOH}$  gave a smaller yield than a direct extraction of the wet macerated material. The resinous extract hardens on standing and smells like tobacco. This crude material could not be crystallized, but was finally obtained as well crystallized yellow-green bricks or prisms from supersaturated petroleum ether (b. 45–50°C.). This aporeine  $\text{C}_{18}\text{H}_{18}\text{O}_2\text{N}$ , m.p. 88–9° to a fluorescent liquid which browns at 225° in the air, but not even at 280–90°

in  $H_2$  or  $CO_2$  in which it may be distilled; it is quite soluble in most organic solvents; in boiling petroleum ether it gives an 11 per cent. solution and at  $15-20^\circ$  is saturated with 3.5 per cent. Aporeine as well as its hydrochloride give the colour reactions of the alkaloids. The blue fluorescence is similar to that of quinine salts and is especially evident in Mg light. The acetate in NaBr gives the hydrobromide, yellowish pearly scales, discolours  $190^\circ C.$ , becomes greenish black  $210^\circ C.$ , m. with decomposition at a high temp. In  $H_2C_2H_3O_2$  aporeine with KI gives the hydriodide as a white amorphous precipitate, decomposes  $200-10^\circ C.$ , m.p.  $250-5^\circ$ . With the calculated amount of  $H_2SO_4$ , aporeine dissolves in  $H_2O$  at  $50-60^\circ$  and on cooling the neutral sulphate separates as fine filaments, which are unstable in air and light. The nitrate obtained similarly is more stable. With the calculated amount of  $H_2C_2O_4$  the acid oxalate is obtained as white tablets, m.p.  $89-90^\circ$ . The acid malate, prisms, yellows  $180^\circ$ , m.p.  $198^\circ$  (gas); the acid tartrate, yellows at  $175^\circ$ , m.p.  $190^\circ$  (decomp.); the acid citrate, needles, m.p.  $81-2^\circ$ , were obtained similarly. The benzoate and salicylate were also obtained as resins. The compound previously named aporeidine was found to be formed from aporeine by the action of light and air and is not an alkaloid related to aporeine as was suspected at first.

**Alkaloids of *Aspidosperma Quebracho*.** A. J. Ewins. (*Trans. Chem. Soc.*, 1914, **105**, 2738) The bark of *Aspidosperma Quebracho* (*quebracho blanco*) yielded 0.06 to 0.2 per cent. of the alkaloid aspidospermine,  $C_{22}H_{30}O_2N_2$ , crystallizing in needles from EtOH, m.p.  $208^\circ C.$ ,  $[\alpha]_D = -99^\circ$  in EtOH and  $-93^\circ$  in  $CHCl_3$ . It is only feebly basic and gives no crystalline salts. When boiled with HI one methoxyl and one acetyl group are hydrolyzed, giving a new base, aspidosine,  $C_{19}H_{26}ON_2$ , crystallizing from EtOH or xylene in rectangular prisms or plates, m.p.  $224^\circ-245^\circ C.$ ,  $[\alpha]_D$  about  $-16^\circ$ ; this gives a hydriodide, m.p. above  $280^\circ C.$ , crystallizing from hot water in octahedra and cubes. Aspidospermine on boiling with dilute hydrochloric acid is converted into deacetylaspidospermine,  $C_{20}H_{26}ON_2$ , m.p.  $110^\circ-111^\circ C.$ ,  $[\alpha]_D +2.8^\circ$ , which on acetylation is reconverted into aspidospermine and on benzoylation gives benzoyldeacetylaspidospermine, m.p.  $186^\circ-187^\circ C.$  Deacetylaspidospermine warmed for a few moments with methyl iodide is converted into a substance,  $C_{20}H_{28}ON_2 \cdot 2CH_3I$ , crystallizing from methyl alcohol

in octahedra, m.p.  $176^{\circ}$ – $177^{\circ}\text{C}.$ , whilst with acetic acid and sodium nitrite it gives a substance,  $\text{C}_{20}\text{H}_{26}\text{O}_4\text{N}_4$ , probably nitronitrosodeacetylaspidospermine, which forms pale yellow prisms, m.p.  $155^{\circ}$ – $156^{\circ}\text{C}.$  with decomposition. Aspidospermine oxidized with chromic acid gives a new base of probable formula,  $\text{C}_{15}\text{H}_{24}\text{O}_2\text{N}_2$ , m.p.  $192$ – $193^{\circ}\text{C}.$ , having a crystalline hydrochloride, m.p.  $286^{\circ}$ – $287^{\circ}\text{C}.$  The existence of the various bases described by Hesse (*Y.B.* 1881, 29), with the exception of aspidospermine and quebrachine (yohimbine), could not be confirmed.

**Berberine, Determination of, as Picrolonate.** E. Richter. (*Archiv. Pharm.*, 1914, 252, 192.) Berberine is quantitatively precipitated from aqueous solutions by an excess of caustic alkali solution. The precipitate is completely soluble in  $\text{Et}_2\text{O}$  and from the  $\text{Et}_2\text{O}$  solution, the alkaloid is reprecipitated by picrolonic acid. 2.5 Gm. of the coarsely powdered drug is extracted with alcohol in a Soxhlet apparatus, the alcoholic solution is evaporated, the residue rinsed with three portions each of 5 c.c. of water into a bottle, mixed with 10 c.c. of NaOH solution (15 per cent.) and 60 Gm. of  $\text{Et}_2\text{O}$  and the mixture is shaken well for 15 minutes. After the addition of 1 Gm. of tragacanth and renewed shaking, 24 Gm. of the clear ethereal liquid, equivalent to 1 Gm. of drug, is decanted, shaken with 5 c.c. of an approximately N/10 picrolonic acid solution, the precipitate collected in a Gooch crucible and, after washing with 5 c.c. of a mixture of one part of  $\text{Et}_2\text{O}$  and two parts of EtOH, dried to constant weight at  $110^{\circ}\text{C}.$  The weight multiplied by 56.1 gives the percentage of berberine in the drug. (See also *Gen. Index.*)

**Brucine Micro-reactions.** J. Scott. (*Chem. and Drug.*, 1915, 86, 144.) The best source of brucine is the false *Angostura* bark, in which the proportion of strychnine to brucine is much less than in any other plant. Pure brucine, such as was used in the following experiment, is a white granular powder, the particles of which present, under the microscope, semi-crystalline shapes. The majority are opaque, while the remainder are semi-transparent, and definitely scored lengthways, the segments readily separating and dissolving apart from one another when the brucine is lying in water. Brucine melts at  $150^{\circ}\text{C}.$  into a pale yellow fluid. The film formed in this way on a glass slide cracks during cooling into characteristic figurings representing

branches and twigs of leafless trees. Many resins behave somewhat similarly, but there appear to be details of this splitting in regard to brucine which call for further consideration, because it generally presents a uniformity of design instead of the irregular criss-crossing noticeable in connection with other melted substances. It is possible to produce minute crystals by only partially melting the substance and then allowing it to cool. In such a case tufts of needle-crystals occur in the semi-transparent portions. Upon adding distilled water to the normal granules of brucine, minute needles begin to form all over them, apparently shooting out rapidly from their surfaces.

A very useful test for the presence of brucine is to add to the substance in strong EtOH a little methyl iodide, whereupon groups of rosetted needle-tufts of methyl brucine iodide will occur. Effects of like formation result when an alcoholic solution of iodine (or else hydriodic acid with iodine) is used on an alcoholic solution of brucine. Should strychnine be present in the alcohol it does not at all interfere with the result. In a solution of brucine acetate treated with AmOH minute needle-crystals are precipitated. It should be pointed out that excess of AmOH fails to yield a precipitate in solutions of brucine, whereas a precipitate occurs when such an excess operates on solutions of strychnine.

Both the carbonated and caustic alkalies precipitate brucine from its solutions. Neither  $K_2CrO_4$  or  $K_2Cr_2O_7$  give a precipitate in solutions of the neutral salts of brucine, but they give precipitates in solutions of the corresponding salts of strychnine. Precipitates of brucine are generally yolk-yellow in colour. Brucine neutralizes acids, and forms a series of salts, most of which are crystalline. The acetate is, however, non-crystalline, although strychnine acetate yields crystals.

The moment  $HNO_3$  touches brucine-powder the latter becomes an intense scarlet colour, which changes to blood-red, yellow-red, and then to yellow. If a drop of water containing some semi-dissolved granules of brucine be touched with a little  $HNO_3$  the solution will become yellow, and transparent rectangular prisms and tablets, often grouped together in rosettes, will quickly rise. Later on the usual red colour will be manifested. It has already been mentioned that oxalic acid and kakotelin result from the test. These reactions can be best studied by placing the brucine in a test-tube, and adding nitric acid of specific gravity 1.4 to it. It is advisable to stand the tube in

cold water meantime. The characteristic red colour will reveal itself, and on diluting the solution freely with water, fluffy yellow specks of kakotelin will separate. These are insoluble in water, but are soluble in dilute acids, and are best crystallized out of dilute  $\text{HNO}_3$  or  $\text{HCl}$ .

After the separation of the kakotelin the remainder should be neutralized with ammonia, and then  $\text{CaCl}_2$  added to throw down the acid as a  $\text{CaC}_2\text{O}_4$ . Another test is to add  $\text{HNO}_3$  to an aqueous solution of  $\text{HgNO}_3$ , and then add this compound to the solution of brucine salt, and warm all. A beautiful carmine tint will occur.

Solutions of brucine are coloured red by chlorine, which can be changed to yellow by means of ammonia.

**Caffeine and Antipyrine, Determination of Mixtures.** W. O. Emery and S. Palkin. (*J. Ind. Eng. Chem.*, 1915, 7, 519.) Transfer to a 150 c.c. separator, by means of two 5 c.c. portions of pure alcohol-free  $\text{CHCl}_3$ , followed by 10 c.c. water, about 0.25 Gm. of the caffeine-antipyrine mixture, add 1 Gm. of  $\text{NaHCO}_3$  and 10–15 c.c.  $\text{N}/2 \text{I}$ , the latter reagent being added in successive small portions and the liquid vigorously shaken for 15–20 seconds after each addition. When all the  $\text{N}/2 \text{I}$  has been thus added, a decided excess of this reagent should be apparent in the liquid after a final vigorous shaking for 1 minute, in which event all the antipyrine will have been converted into the moniodo-derivative. If, however, all the iodine should appear to have been used up, a little more must be added to ensure excess and the mixture again shaken. Now discharge the uncombined iodine by means of a small crystal of hypo, add 15 c.c. pure  $\text{CHCl}_3$ , then shake vigorously 1 minute. After clearing, draw off the solvent into a second separator containing 5 c.c. of water, shake out and after clearing filter the  $\text{CHCl}_3$  into a tared vessel; repeat the extraction with 2 more portions of  $\text{CHCl}_3$  of 25 c.c. each, treating each as before. Evaporate the bulked  $\text{CHCl}_3$  solution, dry the residue at  $105^\circ \text{C}$ . and weigh as caffeine and iodoantipyrine. Dissolve the weighed residue in 5 c.c. of glacial  $\text{HC}_2\text{H}_3\text{O}_2$ , add 10 c.c. of saturated aqueous solution of  $\text{SO}_2$ , transfer to a large beaker, make up to 200 c.c. Add  $\text{AgNO}_3$  solution sufficient to precipitate all the  $\text{I}$ , acidify with  $\text{HNO}_3$ , collect the  $\text{AgI}$ , wash with hot water, then with  $\text{EtOH}$ , dry at  $110^\circ \text{C}$ . and weigh. The weight  $\times 0.8012$  gives the equivalent of antipyrine. The amount of caffeine is found by

multiplying the weight of  $\text{AgI}_2$  by 1.3374, which gives the equivalent of iodoantipyrine. This is deducted from the weight of the  $\text{CHCl}_3$  residue previously obtained. The difference is caffeine.

**Cocaine, Reaction for.** F. Pisani. (*Rend. Soc. Chim. Ital. ; L'Union pharm.*, 1915 [3], 27, 48.) On heating the alkaloid, or its hydrochloride, with a few drops of  $\text{H}_2\text{SO}_4$  containing 2 per cent. of formalin solution a red colour more or less intense, according to the temperature, and a greyish precipitate are formed. Of all the alkaloids in common use only papaverine gives a red colour with this reagent ; but this passes to brown, then to orange yellow. (See also *Y.B.*, 1905, 61 ; 1910, 21 ; 1913, 15, 16, and *Gen. Index*.)

**Coffee, The Determination of Caffeine in.** G. Fendler and W. Stueber. (*Berlin Z. Nahr.-Genussm.*, 28, 9-20.) After grinding raw or roasted coffee so as to pass through a 1 mm. mesh sieve, 10 Gm. of the powder is placed in a glass stoppered bottle with 10 Gm. of 10 per cent.  $\text{NH}_4\text{OH}$  and 200 Gm.  $\text{CHCl}_3$ , and shaken hard and unceasingly for half an hour. Filter the contents of the flask on a folded filter large enough to hold the whole contents of the flask, covering the funnel with a watch glass. 150 Gm. of the filtrate is evaporated in a wide-mouthed 250 c.c. flask on a water-bath, the last traces of  $\text{CHCl}_3$  being removed by a blast of air. The residue is covered with 80 c.c. of hot water and heated on a boiling water-bath, with frequent shakings for 10 minutes, then cooled. To this solution is added, in the case of roasted coffee 20 c.c. (in the case of raw coffee 10 c.c.) of 10 per cent.  $\text{KMnO}_4$  solution and allowed to stand at room temperature for 15 minutes. Excess of  $\text{KMnO}_4$  is removed by the addition of 3 per cent.  $\text{H}_2\text{O}_2$  which contains about 1 c.c.  $\text{AcOH}$  to every 100 c.c. Add first 2 c.c. of the  $\text{H}_2\text{O}_2$ , if still red add another c.c. When the solution is no longer red the flask and its contents are placed on a boiling water-bath and portions of  $\frac{1}{2}$  c.c. of the  $\text{H}_2\text{O}_2$  are added until there is no further change of colour in the solution. This is then heated for 15 minutes on the water-bath, cooled, and filtered on a moistened 9 cm. filter paper. The flask and filter are washed with cold water. The clear filtrate of about 200 c.c. is shaken out with 50 c.c. of  $\text{CHCl}_3$ , then three times with 25 c.c. portions of  $\text{CHCl}_3$ . The combined extracts are evaporated in a wide-mouthed 250 c.c. flask, the last traces being removed by the air blast. The residue of caffeine is dried at  $100^\circ\text{C}$ . to constant weight. (See also *Y.B.*, 1911, 18.)



**Colchicine, Further Investigation of.** A. Windhaus. (*Sitzb. Heidelberg. Akad. Wiss.*, 1914 [18]; *Chem. Abstr.*, 1915, 9, 1481.) The acids formed by the action of  $\text{KMnO}_4$  on colchicine; the behaviour of colchicine with fused  $\text{KOH}$ , and the action of  $\text{I}$  and  $\text{KOH}$  on colchicine are detailed. (See also *Y.B.*, 1914, 6.)

**Cusparine, Isomerization and Decomposition of.** J. Troeger and W. Mueller. (*Arch. Pharm.*, 1914, 252, 459.) Cusparine,  $\text{C}_{18}\text{H}_{14}\text{NO}_2\cdot\text{OCH}_3$ , m.p.  $93^\circ\text{C}$ ., is a quinoline derivative. By the action of moist  $\text{Ag}_2\text{O}$  or  $\text{KOH}$  on its methiodide, ethiodide, or propiodide, isocusparine,  $\text{C}_{18}\text{H}_{14}\text{O}_3\cdot\text{NCH}_3$ , m.p.  $194^\circ\text{C}$ ., is produced. When cusparine is heated in a current of dry  $\text{HCl}$ , pyrocusparine,  $\text{C}_{18}\text{H}_{15}\text{NO}_3$ , m.p.  $255^\circ\text{C}$ ., is formed. This yields a nitro-derivative,  $\text{C}_{18}\text{H}_{12}\text{NO}_2(\text{NO}_2)$ , m.p.  $283^\circ\text{C}$ ., when treated with dilute  $\text{HNO}_3$ . (See also *Y.B.*, 1914, 3.)

**Gelsemium Alkaloids.** A. E. Stevenson and L. E. Sayre. (*J. Amer. Pharm. Assoc.*, 1915, 4, 61.) Besides gelsemine and gelseminine, gelsemium root is found to contain another base, provisionally named *sempervirine*, which is crystalline and gives crystalline salts. It is precipitated from neutral solutions by adding  $\text{HCl}$ ,  $\text{HNO}_3$  or  $\text{H}_2\text{SO}_4$ . The nitrate is specially insoluble: after precipitating it by adding  $\text{HNO}_3$  to the aqueous solution of the hydrochloride, the filtrate will give only a slight opalescence with Mayer's solution. The nitrate is slightly soluble in alcohol from which it separates in well defined yellow needles. The base, liberated from the nitrate, gives a yellow solution with  $\text{CHCl}_3$  and forms pale yellow crystals. The amount of the base obtained being small, a further lot of the drug is being examined with a view to confirming the above, and for further investigation. (See also *Y.B.*, 1913, 21.)

**Heroine Hydrochloride and Diacetyl-Morphine Hydrochloride, Composition and Assay of.** R. T. Harris and A. M. Clover. (*J. Amer. Pharm. Assoc.*, 1915, 4, 291.) Heroine hydrochloride is not, as stated by the manufacturers, the anhydrous salt of diacetylmorphine, but contains 1 mol. of water of crystallization. Commercial diacetyl-morphine hydrochloride is found to vary in its degree of hydration, some specimens containing 1 mol.  $\text{H}_2\text{O}$ , while the others were anhydrous. For the estimation of the alkaloid in its salts the following method is recommended:—An amount of the preparation representing at least

0.10 Gm. of the alkaloid and contained in 10 c.c. of solution is treated with 20 c.c. of  $\text{CHCl}_3$  and sufficient 10 per cent.  $\text{AmOH}$ . to render it slightly alkaline. After shaking vigorously, the  $\text{CHCl}_3$  is drawn off into a container suitable for titrating. The extraction with  $\text{CHCl}_3$  is repeated three times, after which the combined  $\text{CHCl}_3$  extract is evaporated. Add 5 c.c. of  $\text{N}/10 \text{ HCl}$  or sufficient to completely dissolve the alkaloidal residue, then add a few drops of cochineal solution and titrate the excess of acid with  $\text{N}/50 \text{ NaOH}$ .

**Heroine, Determination of Small Quantities of.** R. Miller. (*Amer. J. Pharm.* 1915, **87**, 248.) The following method was used and found, in the absence of morphine or any other interfering substance, to be rapid and sufficiently accurate. A weighed amount of the powder is taken sufficient to contain from  $1/50$  to  $1/20$  grain of heroine. This can be roughly determined by the qualitative reactions. It is placed in a Nessler tube, and 1 c.c. of a 1 per cent. solution of  $\text{H}_2\text{SO}_4$  is added, and then 3 c.c. of a solution of 600 c.c. of commercial  $\text{H}_2\text{SO}_4$ , 300 c.c. of water, and 25 c.c. of a 40 per cent. formaldehyde solution. This reagent will produce a coloration, varying from a yellowish straw for  $1/150$  grain to a deep cherry red for  $1/5$  grain of heroine, depending upon the length of time the reaction is allowed to proceed and the amount of heroine present. A series of standard tubes are prepared, containing  $1/50$ ,  $1/40$ ,  $1/30$ ,  $1/20$ , and  $1/15$  grain of heroine respectively or any other suitable quantity from  $1/150$  grain to  $1/5$  grain, and each of which is treated with the reagent in the same manner and at the same time as the sample. The reaction is allowed to proceed for 10 or 15 minutes in all the tubes, when the coloration in the tube containing the sample is compared with the colorations in the standard tubes.

If, however, a mixture of cocaine and heroine is submitted, the heroine and cocaine as follows is determined: The substance is extracted in the regular manner by the immiscible solvents, and the residue of heroine and cocaine is weighed, and dissolved in a known amount of 1 per cent.  $\text{H}_2\text{SO}_4$ , so that 1 c.c. of the solution will contain between  $1/100$  and  $1/20$  grain of heroine. One c.c. of this solution is put into a Nessler tube and treated with 3 c.c. of formaldehyde sulphuric acid solution. The colour produced is compared and measured as described. The colour standard thus indicates the amount of heroine in

the weighed residuc, and the difference between the heroine and weight of residuc obtained by the immiscible solvent represents the cocaine. A difference of 1/450 grain of heroine will afford an appreciable change of colour when a total of 1/50 grain is present ; and a difference of 1/100 grain can be readily indicated with 1/20 grain under determination.

**Hyoscyamus Muticus as a Source of Hyoscyamine.** (*Bull. Imp. Inst.*, 1915, 13, 29.) Attention is again directed to this Egyptian plant as a valuable source of hyoscyamine which is easily extracted in a crystalline state. The yield is over 0.5 per cent. (See also *Gen. Index* and *Y.B.*, 1905, 97 ; 1908, 95, 238.)

**Immiscible Solvents in the Determination of Aconitine, Codeine, Cocaine, Morphine, and Strychnine.** J. W. Marden and V. Elliott. (*J. Ind. and Eng. Chem.*, 1914, 6, 928.)  $\text{CHCl}_3$  is a much better solvent for aconitine than  $\text{Et}_2\text{O}$ , which is recommended for the purpose by the U.S.P. Three extractions, each with 10 c.c. of  $\text{CHCl}_3$ , will extract more than 99.9 per cent. of the aconitine from 50 c.c. of an ammoniacal aqueous solution. Six similar washings with  $\text{Et}_2\text{O}$  are necessary to extract even 99 per cent.  $\text{CHCl}_3$  is an even better solvent for codeine, two washings sufficing to remove practically 99.9 per cent., using the quantities stated above. For the extraction of codeine,  $\text{Et}_2\text{O}$  is quite useless, the alkaloid being almost equally soluble in wet  $\text{Et}_2\text{O}$  and in water saturated with  $\text{Et}_2\text{O}$ . On the other hand,  $\text{Et}_2\text{O}$  is nearly as good a solvent for cocaine as  $\text{CHCl}_3$  is for codeine, two washings sufficing to extract 99.8 per cent., using the quantities stated above. Two washings with  $\text{CHCl}_3$  will remove this percentage of strychnine from aqueous solution, whereas mixtures of  $\text{Et}_2\text{O}$ - $\text{CHCl}_3$ , recommended by many authors, are much less efficient. In the case of morphine it is suggested that, if the distribution co-efficient for morphine between water or a saline solution and some immiscible solvent under closely defined conditions were accurately determined, it would suffice to work with measured volumes, determine the morphine in an aliquot portion of the immiscible solvent, and calculate that in the remainder of the immiscible solvent as well as that remaining in the aqueous layer. But the determinations of the distribution ratios for morphine are not sufficiently concordant to permit a method to be recommended based on this principle.

It is said that "terpeneless" lemon extract is made by extract-

ing citral from lemon-oil by means of 45 per cent. alcohol. It is shown that alcohol of this strength is a poor solvent for citral compared with the residual terpenes, and that a large quantity of alcohol is necessary to extract 75 per cent. of the citral, even if applied in successive small portions. Alcohol of 50 per cent. strength is shown to be three times as good a solvent for citral.

**Lloyd's Reagent, Behaviour of, with Strychnine and Morphine.**

H. M. Gordin and J. Kaplan. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1656.) Although Lloyd's reagent (*Y.B.*, 1914, 1, 11) completely removes morphine and strychnine from aqueous solutions, the bases cannot be recovered quantitatively from the dried precipitates. In the case of morphine, the amount of base extracted by means of MeOH was only 90 per cent. of that originally present. With the strychnine precipitate, even 10 successive extractions with  $\text{CHCl}_3$  failed to remove all the alkaloid. Lloyd's reagent is not available, therefore, for the quantitative determination of these alkaloids.

The precipitate obtained with Lloyd's reagent from the solution of a strychnine salt is almost tasteless, although it contains all the alkaloid originally present in solution. It has, however, all the physiological action of strychnine diluted by an inert substance so that the union of the alkaloid and reagent is disrupted in the living digestive apparatus. Yet, *in vitro*, pepsin in dilute HCl does not remove any strychnine from the strychnine precipitate obtained with Lloyd's reagent, nor have trypsin nor ptyalin any dissociating effect on the compound. (See also *Y.B.*, 1914, 1, 11.)

**Morphine and Phenols, Colour Test for, with Uranium Salts.**

J. Aloy and C. Rabaut. (*Bull. Soc. Chim.*, 1914, 15, 680.) A few drops of saturated solution of  $\text{UO}_2$ ,  $2\text{NO}_3$ , or of  $\text{UO}_2 \cdot 2\text{C}_2\text{H}_3\text{O}_2$  added to a perfectly neutral solution containing morphine will give a red colour. When only minute traces of morphine may be present, the reagent should be added to the dry alkaloidal residue. The test will detect 0.00005 Gm. of morphine. A similar reaction is given by other substances containing a free phenolic group. (See also *Gen. Index* and *Y.B.*, 1905, 61; 1909, 59; 1911, 24.)

**Morphine Bases in which Acetic Anhydride does not cause the Fission of the Nitrogen Ring.** M. Tiffeneau. (*Bull. Soc. Chim.*, 1915, 17, 109.) Diacetylmorphine has been prepared

by warming morphine hydrochloride with acetic anhydride on a water-bath for 20 hours; m.p.  $173^{\circ}\text{C}.$ ; b.p.  $272^{\circ}\text{--}274^{\circ}\text{C}.$  under 22 mm. On further treatment with acetic anhydride for several hours at  $170^{\circ}\text{--}180^{\circ}\text{C}.$ , it undergoes no further change, therefore fission of the nitrogen ring does not occur. Codeine, ethylmorphine, and thebaine (prepared by reduction of thebaine with  $\text{SnCl}_2$ ) can be acetylated in a similar manner, and the acetyl derivatives, like diacetylmorphine, are unaffected by further treatment with acetic anhydride at  $170^{\circ}\text{--}180^{\circ}\text{C}.$  Acetylcodeine melts at  $133^{\circ}\text{C}.$ , and boils at  $258^{\circ}\text{C}.$  under 11 mm. Acetylmorphine melts at  $131^{\circ}\text{C}.$ , and boils at  $260^{\circ}\text{--}262^{\circ}\text{C}.$  under 12 mm. pressure.

**Morphine and its Salts, New Test for.** T. H. Oliver. (*Med. Chron.*, 27 [4]; *Chem. Abstr.*, 1914, 8, 3314; *J. Am. Med. Assoc.*, 68, 513-4.) To a solution of morphine add a few c.c. of  $\text{H}_2\text{O}_2$  and a little strong  $\text{AmOH}$ ; stir the mixture with a piece of Cu wire and the previously colourless solution assumes a port wine tint, the reaction being accompanied by a considerable evolution of gas. The test is very delicate, 0.02 mg. being readily shown. In applying the test to minute traces, KCN solution is added at the end of the reaction to remove any blue colour produced by the Cu. The addition of KCN before stirring with Cu tends to prevent the occurrence of the reaction. The reaction is probably dependent upon a combination of all four reagents, the Cu acting as a catalyst.  $\text{MeNH}_2$  may be used in place of  $\text{AmOH}$  but not  $\text{Me}_2\text{NH}$  nor  $\text{NaOH}$ . The  $\text{NH}_2$  group appears essential to the reaction. Methyl- and ethyl-morphine and apomorphine do not give the reaction. Twelve of the more common alkaloids gave negative results with the test. Other substances as catalysts gave negative results. The red substance formed is insoluble in the ordinary morphine solvents, also in  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$  and  $\text{C}_6\text{H}_5\text{CH}_3$ . The whole of the morphine is apparently changed, as none of the alkaloid is precipitated from the ammoniacal solution.

**Morphine in Pills or Tablets, Determination of.** J. B. Williams. (*Amer. J. Pharm.*, 1914, 86, 310.) A number of pills or tablets, or a quantity of the sample for assay containing not more than 0.5 Gm. morphine (preferably from 0.1 to 0.2 Gm.), is dissolved in a few c.c. of acid water, in a separator, keeping the volume of the liquid as small as possible (from 5 to 10 c.c.); add from 15 to 25 c.c. of mixture of  $\text{EtOH}$  1 part and  $\text{CHCl}_3$

2 parts by volume and 2 or 3 c.c. of 10 per cent. solution of AmOH, or sufficient to make distinctly alkaline. Shake well for 2 or 3 minutes. After separation, draw off the  $\text{CHCl}_3$  solution, filter through cotton, well wetted with  $\text{CHCl}_3$ , into a wide-mouth flask or beaker of about 150 c.c. capacity. Repeat the extraction with two further like portions of the EtOH- $\text{CHCl}_3$  mixture and then with three 10 c.c. portions of  $\text{CHCl}_3$ . Evaporate the EtOH- $\text{CHCl}_3$  solution on a water-bath under a current of warm air to dryness, add a few c.c. of EtOH and again evaporate. Dissolve the residue in an excess of N/10 acid and titrate back with N/50 alkali, using cochineal as indicator. Each c.c. of acid neutralized by the alkaloid = 0.0301 Gm. of crystalline morphine or 0.0376 Gm. morphine sulphate. (See also *Y.B.*, 1914, 13, 14.)

**Morphine Nitrate and Acetate, Purity of, in American Commerce.** H. Engelhardt and O. E. Winters. (*J. Amer. Pharm. Assoc.*, 1915, 4, 289) It is desirable that official standards should be given for the morphine content of these salts, the purity of which, as occurring in American commerce, is not satisfactory. Morphine nitrate rarely exceeds 90 per cent. of pure salt. The purity of the acetate is far below 100 per cent., which is noteworthy since most samples give off the odour of acetic acid and therefore should be more basic than the normal salt. Comparative assays with the U.S.P., isobutyl-alcohol-chloroform, Vanderkleed, Buchbinder, Puckner and Schaefer methods are quoted, also the nitron method for the nitrate.

**Morphine, Stability of, in Presence of Putrefaction.** F. Doepmann. (*Chem. Ztg.*, 1915, 39, 69; *Chem. Abstr.*, 1915, 9, 1663.) Separate quantities of 1 kil. of chopped, lean horse flesh were mixed with 200, 100, 50 and 20 Mgm. respectively of morphine-hydrochloride and 200 Gm. of the mixture investigated after 1,  $2\frac{1}{2}$ ,  $5\frac{1}{2}$ , and 11 months. The putrefying mass was thoroughly extracted with very dilute AcOH, first cold, then warm, and finally on the water-bath. The acid extract was concentrated, precipitated with EtOH, the EtOH-free filtrate precipitated with  $(\text{AcO})_2\text{Pb}$ , excess of Pb removed by  $\text{H}_2\text{S}$  and the solution concentrated, made alkaline with  $\text{NH}_4\text{OH}$ , and extracted repeatedly with warm  $\text{CHCl}_3$ . The residue from the  $\text{CHCl}_3$  extract was dissolved in dilute  $\text{H}_2\text{SO}_4$  and extracted with pure amyl alcohol to remove colouring matters, then made alkaline with NaOH and extracted with a small amount of  $\text{CHCl}_3$  to remove ptomaine bases, and finally made alkaline with

$\text{NH}_4\text{OH}$  and repeatedly extracted with warm  $\text{CHCl}_3$ . The pale yellow varnish left on evaporating the  $\text{CHCl}_3$  gave in every case the characteristic reactions of morphine.

**Morphinometric Assay of Opium, The Lime Method for.** A. B. Lyons. (*J. Amer. Pharm. Assoc.*, 1915, 4, 92.) Of the numerous methods that have been proposed for the morphinometric assay of opium, two only seem to have found favour in national Pharmacopœias. In one, the drug is exhausted with water, the solution concentrated to a small volume, alcohol and ether added together with water of ammonia, the mixture shaken well and allowed to stand for a specified time for separation of the morphine in crystals. In the other, the powdered opium is mixed with lime and a certain proportion of water, allowed to macerate, with occasional stirring during half an hour, the solution filtered and an aliquot portion of the filtrate treated with  $\text{AmCl}$  which causes the morphine to separate in crystalline form. The advantages claimed for the second method are (1) rapidity of execution; (2) superior purity of the morphine obtained, owing partly to the fact that lime combines with morphine forming a very soluble compound—a property not shared with it by narcotine or most of the other alkaloids of opium—partly because the lime throws out of solution certain organic acids and other compounds which otherwise are liable to be thrown down with the morphine; (3) alleged uniformity of results.

Against the lime method it is urged: (1) It involves unavoidably the principle of the aliquot part; (2) crystallization of the morphine is from a more dilute solution than in the first general method, hence more of the morphine is held in solution so that an arbitrary correction is generally prescribed to compensate this loss (It is generally admitted that there is also loss of morphine in the first assay method, in which no correction factor is generally prescribed); (3) the assay requires that the opium be in the form of a powder, whereas opium is imported in a moist condition, so that it must be dried (the loss of weight noted) and reduced to a powder—operations which greatly lengthen the time required for an assay.

Discussing both methods, the author concludes in favour of the lime method. It is pointed out that by the use of reasonable care accurate results are obtainable. (See also *Gen. Index* and *Y.B.*, 1904, 118, 119, 121; 1905, 120, 121; 1906, 50, 55; 1907, 116; 1908, 150; 1912, 30, 31; 1913, 12, 22, 358, 359; 1914, 16.)

**Opium and Preparations, Morphinometric Assay of.** A. R. L. Dohme. (*J. Amer. Pharm. Assoc.*, 1915, 4, 85.) The following shaking-out method is recommended as being more accurate than a lime process or a crystallizing method.

*Powdered Opium.*—Four Gm. of the powdered opium is macerated with water in the usual way either by allowing to stand over night or by shaking for three hours, then exhausting the opium with water and evaporating the combined filtrate and wash-water to about 50 c.c. The solution is then transferred to a separator, made decidedly alkaline with KOH or NaOH solution which holds the morphine in solution as alkali morphinate. The solution is then shaken out with several portions of 20 c.c. each of  $\text{Et}_2\text{O}$  in order to remove the alkaloids other than morphine. The alkaline solution is then acidified with  $\text{H}_2\text{SO}_4$ , made again *slightly* alkaline by the addition of  $\text{AmOH}$ , and is then extracted by shaking out with several portions of a mixture of equal volumes of  $\text{CHCl}_3$  and isobutyl-alcohol, until all the alkaloid is removed. The isobutyl-alcohol- $\text{CHCl}_3$  solutions are then filtered into a distilling flask, the filter paper washed with more solvent, the  $\text{CHCl}_3$  distilled off under ordinary conditions and the isobutyl-alcohol subsequently under diminished pressure. If the original mixture is distilled under diminished pressure bumping very frequently takes place. The residue in the flask is taken up in an excess of standard acid; the acid solution is well diluted with water, and, after the addition of a few drops of methyl red, the excess of acid is titrated back with standard alkali. From the amount of acid used, the morphine is calculated.

For the assay of *Gum Opium* the following modification should be used: 20 Gm. of opium representing an average sample of as many balls is exhausted in the regular way by means of water, the combined aqueous liquids are made up with sufficient water to obtain 500 c.c., and 100 c.c. of this solution are subjected to the assay process. Thus a fairly representative sample of the opium can be obtained.

*Tincture of Opium.*—From 25 c.c. of the tincture, the alcohol is expelled by heating; the residual liquid acidulated with sulphuric acid, the solution filtered into a separator and the filter and residue washed with small portions of water until the combined filtrate and wash-water measure 50 c.c. This is then treated as the concentrated aqueous extract in the powdered opium assay.



**Concentrated Fluid Extract of Opium.**—Five c.c. of fluid extract is freed from EtOH by evaporation; the residual liquid is rendered acid with  $\text{H}_2\text{SO}_4$  and filtered into a separator. The filter contents are well washed until the total filtrate measures about 50 c.c. The assay is then carried on as described for the tincture.

**Fluid Extract and Camphorated Tincture of Opium.**—The shaking-out method is specially useful for preparations containing a small amount of morphine, such as paregoric. For the fluid extract 25 c.c. are taken, and for the camphorated tincture 50 c.c. The process is practically identical with that given for the tincture. Most of the benzoic acid and all the camphor is evaporated or filtered out. Any trace of camphor left does not interfere with the titration.

**Extract of Opium.**—Five Gm. of extract is dissolved in water and the liquid evaporated to 50 c.c., then treated as directed for tincture.

The method is specially useful in the assay of morphine pills or tablets. By its means morphine can be separated and quantitatively determined from a mixture containing other alkaloids. It is superior in every sense to the lime method and also to the official U.S.P. process. Its inclusion in the future U.S.P. is recommended. (For references to previous notes, see after preceding abstract.)

**Opium, Effect of Moulds on the Alkaloids of.** O. v. F r e d e r i c h s. (*Z. Physiol. Chem.*, 1914, **93**, 276; *Chem. Abstr.*, 1915, **9**, 952.) *Penicillium viridicatum* and *Citromyces glaber* have no effect upon the morphine, narcotine, and codeine content of opium; *Aspergillus niger* does not affect the morphine but attacks narcotine and codeine, while *A. ostianus* affects morphine also, but to a very small extent. The ordinary growth of moulds, as met with in drugs, scarcely affects the alkaloidal content of opium.

**Oxymorphine, Detection of, in Presence of Morphine.** L. Grimbert and A. Leclère. (*J. Pharm. Chem.*, 1914, **10**, 425.) The reaction is based on the insoluble precipitate given by oxymorphine with  $\text{K}_6\text{Fe}_2\text{Cy}_{12}$  in presence of  $\text{Na}_2\text{C}_2\text{H}_3\text{O}_6$  in a neutral solution. The reagents employed are a 1:100 solution of  $\text{K}_6\text{Fe}_2\text{Cy}_{12}$  and a neutral 1:10 solution of  $\text{NaC}_2\text{H}_3\text{O}_6$ . In solutions containing not more than 1:1000 of oxymorphine 2 drops of each reagent are added; for a 1:100 solution 6 drops of

each. When morphine and oxymorphine are supposed to be present together the bases are converted into hydrochlorides, and the test applied to the neutral solution. If no precipitate or turbidity appears, less than 1:20000 of oxymorphine is present. If more than that quantity is present, the precipitate obtained will be proportional to the amount. Morphine, codeine, apomorphine, thebaine, quinine, strychnine, brucine and caffeine give no precipitate under these conditions, and the identity of oxymorphine may be established with any precipitate obtained by the following colour test. To the base, liberated from the ferricyanide precipitate, in a watch glass a drop of formalin— $\text{H}_2\text{SO}_4$  is added. A fine green colour will be produced. This is usually given as the characteristic test for oxymorphine. This, however, is not quite correct. The green colour is due to the presence of a trace of  $\text{K}_3\text{Fe}_2\text{Cy}_{12}$ . If oxymorphine is quite pure the colour will be red. On adding a minute trace of  $\text{K}_3\text{Fe}_2\text{Cy}_{12}$  an intense green colour is produced. In the case of morphine the addition of  $\text{K}_3\text{Fe}_2\text{Cy}_{12}$  does not affect the original purple tint given with the formalin— $\text{H}_2\text{SO}_4$  reagent, and none of the other alkaloids above mentioned give a green colour. Oxymorphine may be further identified in the base liberated from the  $\text{K}_3\text{Fe}_2\text{Cy}_{12}$  precipitate, by converting a little into the hydrochloride, evaporating off  $\text{HCl}$ , and adding a drop of saturated solution of  $\text{Na}_2\text{SO}_4$ . Formation of crystals of the insoluble sulphate of oxymorphine will then be observed. (See also *Gen. Index.*)

**Papaver Orientale, Alkaloids of.** W. Klee. (*Archiv. Pharm.*, 1914, 252, 211.) During the period of most rapid growth, thebaine is the main alkaloidal product. In autumn, when the vegetative process slackens, isothebaine is found to predominate in the root of the plant. The relation of this base to thebaine and the structure of its molecule are discussed at length. It occurs in nearly colourless highly refractive rhombic crystals,  $\text{C}_{19}\text{H}_{21}\text{O}_3\text{N}$ , m.p.  $203^\circ\text{--}204^\circ\text{C}$ .;  $[\alpha]_D +285.1^\circ$ . Its colour reactions, salts and substitution products are described. (See also *Y.B.*, 1911, 36.)

**Papaver Orientale, Secondary Alkaloids of.** J. Gadamér. (*Arch. Pharm.*, 1914, 252, 274–80. The amorphous basic mixture obtained after the separation of thebaine and isothebaine contains two alkaloids without, and at least three with, phenolic properties. In the first group protopine was

detected, while in the second an alkaloid belonging to the glaucine group, provisionally designated *glaucidine*, was isolated. The latter gives colour reactions very like those obtained with glaucine. Its  $[\alpha]_D = 47-54^\circ$ . Attempts to convert glaucidine into glaucine by methylation were unsatisfactory.

**Pareira Root, Alkaloids of.** M. Scholtz and O. Koch. (*Arch. Pharm.*, 1914, **252**, 513; *J.S.C.I.*, 1915, **34**, 680.) Pareira root contains mainly bebeerine, or sometimes  $\beta$ -bebeerine. Bebeerine, isobebeerine, and  $\beta$ -bebeerine are isomeric compounds. Isobebeerine is an isoquinoline derivative and an isomeride of codeine. As the free hydroxyl group is phenolic in character, isobebeerine resembles morphine in its behaviour towards several reagents. Isobebeerine is not a febrifuge, but is a narcotic. (See also *Y.B.*, 1913, 217.)

**Physostigmine, Further Investigation of.** F. Straus. (*Annalen*, 1914, **406**, 332; *Chem. Abstr.*, 1915, **9**, 57.) Eseroline, using the ordinary method for estimation of -NMe, gave values corresponding to 1 Me, but with the Pregl apparatus, the values are nearer those for 2 -NMe. Physostigmol, prepared by heating eseroline-MeI in  $\text{CO}_2$  to  $200^\circ\text{C}$ . for a short time and dried by melting in a vacuum and subliming under 0.01-0.02 mm., gives figures for  $\text{C}_{10}\text{H}_{11}\text{ON}$  and not  $\text{C}_{11}\text{H}_{11}\text{ON}$  as given before; m.p.  $103^\circ$ ; recrystallized samples melt higher but unsharp. During the preparation  $\text{Me}_2\text{NH}$  is split off besides a second compound richer in C. Methyl ether, by shaking the alkaline with  $\text{Me}_2\text{SO}_4$ , leaves from MeOH, m.p.  $60-1^\circ$ ; this shows all the properties of a phenol ether; it contains 1-NMe and 1-OMe group. The picrate forms fine red needles, soluble in EtOH with a yellow colour (dissociation). (See also *Gen. Index* and *Y.B.*, 1913, 33.)

**Pilocarpine, Isolation of, from Cosmetic Preparations.** F. Adam. (*Archiv. Chem. Micros.*, 1914, **7**, 201; *Chem. Abstr.*, 1915, **9**, 1531.) In testing hair preparations for pilocarpine, care should be taken to avoid large excess of caustic alkali. The base should be liberated by means of  $\text{NaHCO}_3$  and shaken out with  $\text{CHCl}_3$ . The latter is a better solvent than  $\text{Et}_2\text{O}$  for the purpose. The quantitative separation of pilocarpine from other alkaloids is not easy.

**Quinine and Urea Hydrobromide and Hydrochloride.** P. G. Golubev. (*J. Russ. Phys. Chem. Soc.*, **46**, 189; *Chem.*

*Abstr.*, 1914, 8, 2549.) The double quinine urea hydrobromide,  $C_{20}H_{24}N_2O_2 \cdot HBr \cdot CH_4N_2O \cdot HBr$ ,  $3H_2O$ , made by the interaction of 1 mol. of  $CO(NH_2)_2$  with 1 mol. of quinine dihydrobromide (yield, 72 per cent.), m.p.  $70-5^\circ C$ ., loses its  $H_2O$  at  $80^\circ C$ . or over  $H_2SO_4$ , and reabsorbs about 2  $H_2O$  on exposure to the air. At  $130-140^\circ C$ . it decomposes into quinine hydrobromide,  $NH_4Br$ ,  $CO_2$ ,  $NH_3$  and  $H_2O$ . The corresponding double hydrochloride was made by a similar method. It contains 3 mols. of  $H_2O$ .

**Strychnine, Colour Test for, with  $MnCO_3$  and  $H_2SO_4$ .** G. Denigés. (*Bull. Soc. Pharm. Bordeaux; Repertoire Pharm.*, 1915 [3], 27, 53.) The violet colour-reaction obtained with Guérin's test (*Y.B.*, 1914, 23) with  $H_2SO_4$  and  $MnCO_3$  is due to a trace of  $MnO_2$  contained as an impurity in the latter. The reaction is essentially one of oxidation. Consequently it cannot occur with a pure manganous salt as stated by Guérin, but may be given by a manganic compound in presence of  $H_2SO_4$ . The author does not agree with Guérin that the Mn reaction is more sensitive than the familiar test with  $H_2SO_4$  and  $K_2Cr_2O_7$ . Also, from the fact that  $MnCO_3$  containing a varying amount of  $MnO_2$ , the reaction cannot be definitely reliable. Moreover, the colour of the reagent, and even more so that of a solution of  $MnO_2$  in  $H_2SO_4$ , is too nearly alike that given with minute traces of strychnine to render the test as useful as the  $K_2Cr_2O_7$  reaction.

**Strychnine, Substances which Mask the Colour Reactions of.** E. Mameli. (*Boll. Chim. Farm.; J. Pharm. Chim.*, 1915, 11, 125.) The presence of the following drugs will vitiate the familiar colour reaction of Otto, with  $H_2SO_4$  and  $K_2Cr_2O_7$ , or of Mandelin with  $H_2SO_4$  and ammonium vanadate—Phenacetin; parphenetidin; para-aminophenol; phenocol; salacetol; protocathecuic acid; amino-ethyl-pyrocatechin; amylochloral; guaiacol; acetylguaiacol; heroine; helmitol; pyramidon; zinc sulphocarbolate; glycerin, and hydrochloric acid. [The majority of these would be eliminated by treatment with  $H_2SO_4$  at  $100^\circ C$ . as in the process of Girdwood and Rogers.—*Ed. Y.B.*]

**Strychnos Henningsii, Alkaloidal Constituent of.** (*Bull. Imp. Inst.*, 1915, 13, 30.) The bark and fruit of the "Hard Pear" tree of South Africa have been examined. The bark contains as much as 5.3 per cent. of a bitter alkaloid which has not been

obtained definitely crystalline. Its salts are also amorphous, except the sulphate, which crystallized with difficulty. The free base is soluble in most organic solvents, except  $\text{Et}_2\text{O}$ , in which it dissolves only slightly; it is insoluble in water. It gives no reactions similar to those of strychnine or of brucine. The kernels of the seeds contain a quantity of alkaloid probably similar to that in the bark. The husks contained only a little of the base.

**Strychnos Nux Vomica Seeds, New Alkaloid, Struxine, from.**  
H. H. Schaefer. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1677.)  
The author has isolated a new base, *struxine*,  $\text{C}_{21}\text{H}_{30}\text{N}_2\text{O}_4$  from the mother liquors obtained in the manufacture of strychnine. It is possible that the new alkaloid is a decomposition or oxidation product of strychnine. Those "beans," which are worm eaten and partly decomposed, contain more of it than do good beans. The average amount found in decomposed beans was 0.1 per cent., but the amount varies considerably in different parcels of "beans." In neutralizing the acid-solution of the raw alkaloidal sulphates of nux vomica, the new alkaloid separates out as a base, when the liquors become just neutral, or are still slightly acid, while strychnine and brucine remain in solution as sulphates. It was found that only few lots of nux vomica beans contained this substance. These were small "beans" from Cochin-China.

The alkaloid, purified by recrystallization from  $\text{EtOH}$ ; consists of colourless crystals containing no water of crystallization. Upon exposure to air and light, however, the crystals gradually develop a faint yellowish colour. No melting point could be determined, as the substance begins to char at about  $250^\circ\text{C}$ . A solution of it gives typical alkaloidal reactions with most reagents. It is only very slightly bitter, which lack of taste is probably due to its extreme insolubility in water, since the more soluble salts of struxine are distinctly bitter, but have not the intensity of either strychnine or brucine, or any of the igasurines, as reported in literature.

The following solubility determinations were made:—In water, 1:5000 c.c.; in  $\text{CHCl}_3$ , 1:1 2.3 c.c.; in alcohol, 1:190 c.c.; in ether, 1:450 c.c.; in methyl, 1:210 c.c.; in benzol, 1:35 c.c.

Upon microscopical examination, it was found that the crystals are all of well-defined rhombic form. Numerous salts of the base are described. Struxine gives no colour with  $\text{H}_2\text{SO}_4$ ; and when

a particle of  $K_2Cr_2O_7$  is added to the acid mixture a yellow colour passing to green is formed. With  $HNO_3$  no colour is formed and the solution on evaporation leaves a yellow residue which turns red with alkalis. A great number of colour reactions are given which differ entirely from those of strychnine, brucine and igasurine.

**Tobacco and Tobacco Preparations in Nicotine, Determination of.** H. B. Rasmussen. (*Chem.-Zeit.*, 1915, **39**, 25; *J.S.C.I.*, 1915, **34**, 300.) Tobacco and tobacco preparations are extracted with 20 per cent. NaOH solution and then with a mixture of  $Et_2O$  and light petroleum. The latter extract, containing the whole of the nicotine, is shaken with dilute HCl, and the nicotine is determined by precipitation with silicotungstic acid, the precipitated salt,  $2C_{10}H_{14}N_2 \cdot 2H_2O \cdot 12W_3SiO_2 + 5H_2O$ , being dried at  $120^\circ C$ . and weighed as  $2C_{10}H_{14}N_2 \cdot 2H_2O \cdot 12W_3SiO_2$ . (See also *Y.B.*, 1914, 14, 15.)

## ANIMAL PRODUCTS.

**Adrenine (Epinephrine) Content of Suprarenal Glands, Variation of.** A. Seidell and F. Fenger. (*U.S. Treasury Dep. Hygienic Lab. Bull.*, **100**, 57.) A very large number of adrenal glands of the ox, hog and sheep have been examined and the amount of adrenine therein determined. Although wide variation in the amount of the base was observed with these animals, this could not be referred to any special season. Ox adrenals were richest, yielding from 1.0 to 0.4 per cent. Hogs' adrenals gave from 0.4 to 0.05 per cent. Sheep's adrenals were poorest, giving 0.3 to 0.05 per cent. Not only were the ox glands twice as rich as those of the sheep, but they were also much larger. Adrenine was determined colorimetrically by Seidell's method. In this, the pink colour formed when the powdered gland suspended in water is mixed with purified pyrolusite powder, is matched against standards obtained by comparing known amounts of pure epinephrine. (See also *Y.B.*, 1913, 2.)

**Animal Charcoal, Tests for the Adsorptive Power of.** Wiechowski. (*Muench. Med. Woch.*; *Schweiz. Apoth. Zeit.*, 1915, **53**, 80.) The Austrian Ministry of the Interior has issued an official test for animal charcoal intended for medicinal use, which originally contained two important printer's errors. The amended test reads as follows. One Ggm. of finely sifted animal

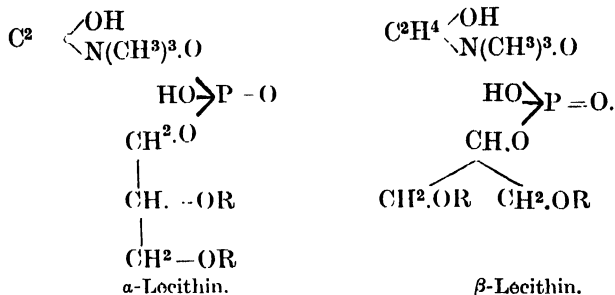
charcoal previously dried at  $120^{\circ}\text{C}$ . should give, when shaken in a closed vessel with 20 c.c. of a 1.5 : 1000 solution of methylene blue hydrochloride, a completely colourless solution, without filtering, in one minute. If 3 Gm. of the same animal charcoal is mixed with 65 c.c. of the above methylene blue solution and drunk, the urine passed during the next 24 hours should show no green colour.

**Erepsin.** S. K o b z a r e n k o. (*Biochem. Z.*, **66**, 344 ; *Chem. Abst.*, 1914, **8**, 3579.) There is a considerable amount of erepsin in the intestinal mucosa, which may play an important rôle in the process of digestion. Erepsin is destroyed at  $58^{\circ}$ , and even at  $50^{\circ}$  if maintained at that temperature for some time. Organic or inorganic acids, many of them in concentration of 0.02 to 0.04 per cent., destroy the enzyme in 1 hour ; weaker acids retard the action without destroying it, while in concentrations of 0.002 to 0.005 per cent. they have no effect. Alkalies have a weaker action than acids ; solutions of 0.06 to 0.13 per cent. destroy the enzyme in 1 hour, while solutions of 0.06 per cent. are indifferent. Na salts decrease the activity, while K salts have no effect. Salts of  $\text{H}_2\text{SO}_4$  and  $\text{H}_3\text{PO}_4$  are indifferent, while salts of other acids decrease the activity. Alanine and glycocol in concentrations of 3-4 per cent. have no effect upon the enzyme ; EtOH retards its activity, it becoming indifferent in a concentration of 2 per cent. The pancreatic juice of man has a small amount of erepsin, that of the dog still less. The liver contains the most erepsin ; then follow the intestinal mucosa and kidneys, while the muscles, spleen and blood serum contain traces. In P poisoning there is an increase of ereptic activity in all the organs. The blood serum appears to increase the activity of the erepsin in the organs, this increase probably being related to the erepsin in the serum.

**Fibrin.** A. W. B o s w o r t h. (*J. Biolog. Chem.*, 1915, **23**, 91 ; *Chem. Abstr.*, 1915, **9**, 810.) Pure ash-free fibrin was obtained by precipitating it from 0.2 per cent. NaOH solution by means of  $\text{HC}_2\text{H}_3\text{O}_2$  0.3 per cent. ; then reprecipitating it four times from NaOH solution 0.05 per cent. It resembles casein in all respects except that it is readily dissolved by dilute  $\text{HC}_2\text{H}_3\text{O}_2$ . It forms definite compounds with both acids and bases. With the latter it combines with 4 equivalents, giving neutral compounds, and also a series of acid salts containing 1, 2, and 3 equivalents of base. The alkali salts are

soluble. It is not strong enough to decompose  $\text{CaCO}_3$ . The molecular weight of fibrin is about 6666.

**Lecithin of Egg Yolk and of Brain Substance.** O. B a i l l y. (*Comptes rend.*, 1915, **160**, 395 ; also *Journ. Pharm. Chim.*, 1915, **11**, 204.) In the egg, lecithin consists of a mixture of at least two isomeric forms,  $\alpha$ - and  $\beta$ -lecithin. These have the following constitution, in which R represents a fatty acid residue :—



Three-fourths of egg lecithin is composed of  $\beta$ -lecithin, the remaining fourth being  $\alpha$ -lecithin.

Since natural lecithin is evidently a mixture, the author questions if the preference shown for definitely crystalline calcium glycerophosphate for use in medicine is justified. Since a mixture of the two forms exists in the organism, logically this mixture should be exhibited in medicine. As far as the assimilability of the two forms, there appears to be no appreciable difference.

**Marine Animal Oils, Colour Reaction for.** T o r t e l l i and J a f f e. (*Schweiz. Apoth. Zeit.*, 1915, **53**, 107.) One c.c. of the oil in a graduated cylinder is mixed with 6 c.c. of  $\text{CHCl}_3$  and 1 c.c. of ice cold  $\text{HC}_2\text{H}_3\text{O}_2$ . Forty drops of a 1:10 solution of Br in  $\text{CHCl}_3$  is then added, the mixture quickly shaken up, and the cylinder stood on white paper. In less than a minute, marine animal oils show a pinkish tint gradually becoming green, the colour deepening for an hour. The purer the animal oil, the more intense is the colour. Hydrated marine animal oils also give a bright emerald green reaction. Vegetable oils remain unaffected for a minute, then show a pale yellow colour which darkens on standing. The fat of terrestrial animals is at first coloured yellow, then brownish, and only shows a slight greenish reflexion in rare cases.



**Milk, Effect of Sodium Citrate in Preventing Curdling of by Rennin.** A. W. Bosworth and L. L. Van Slyke. (*Bull. No. 34, New York Agric. Exp. Stat.*, 1914, 3; *J.S.C.I.*, 1915, 34, 678.) The curdling of milk by rennin is retarded or prevented by addition of sodium citrate. No curdling takes place when the quantity of sodium citrate reaches 1.7 grains per oz., and with smaller quantities, the softness of the curd increases with the quantity of citrate. The effect is due to a reversible reaction between the sodium citrate and the calcium caseinate of the milk, with formation of calcium-sodium caseinate, which latter yields a soluble calcium-sodium paracaseinate by the action of rennin.

**Milk Crystals in Sweetened Condensed Milk.** M. Sato. (*J. Coll. Agr. Tohoku Imp. Univ.*, 5, 321; *Chem. Abst.*, 1914, 8, 3082.) The crystalline bodies in sweetened condensed milk belong to a number of different groups. The largest crystals, which have the greatest number of axes, are milk sugar crystals of the monoclinic system, but lactose may change its shape when the milk is condensed with cane sugar. Oblique, monoclinic crystals of cane sugar were never found except in milks with too little water, and even then with uncertainty.  $\text{Ca}_3(\text{C}_6\text{H}_5\text{O}_7)_2$  forms the major part of the needle-shaped crystals, which occur either isolated or in sheaves. There are also clusters of needle-shaped crystals of tyrosine. From the amorphous sediment,  $\text{Mg}_3(\text{PO}_4)_2$  and  $\text{Ca}_3(\text{PO}_4)_2$  were isolated. Leucine is present in a globular form and cystine in plates. The latter two substances are rare, and appear to be formed under the same conditions as tyrosine. A large number of micro-drawings are given.

**Ovovitellin.** G. Rosmini and O. G. D'Alceo. (*Arch. farm. sci. affn.*, 1914, 3, 29; *Chem. Abstr.*, 1915, 9, 809.) Yolks of eggs separated mechanically from the whites are exhausted with cold EtOH, to remove lecithin and lutein. The residue is pressed to separate the oil and the little EtOH still remaining. It is then extracted with  $\text{Et}_2\text{O}$  and evaporated at 35–40°, the product being ovovitellin, available for alimentary use. It contains water 18.25, substances soluble in water 7.39, ash 3.96, N 12.23, P 9.86 per cent. (0.48 per cent. is organic P); it yields by the action of pepsin, 16.30 per cent. of nuclein. In dogs, 2.5 g. ovovitellin together with the daily reaction, had a favourable influence upon the general condition

of the animal, the activity of exchange, and appreciably increased the weight. Ovovitellin is indicated in all diseases where small doses of substances rich in N and organic P must be given.

**Pepsin, Assay of, with Edestin.** Delauny and Bailly. (*Bull. Sci. Pharm.*, 1915; *Répertoire Pharm.*, 1915, 27, 84.) The vegetable globulin, edestin, gives better and more definite results in testing the proteolytic activity of pepsin, than the dried fibrin used for the purpose in the official test of the French Codex. Edestin is prepared from hemp seed. The seeds are deprived of fat by treatment with petroleum ether. The dried and powdered fat-free residue is macerated in a 1 : 10 solution of NaCl. The liquid is then neutralized with  $\text{Ba}(\text{OH})_2$  and the NaCl removed by dialysis; or the edestin is simply precipitated by adding a large excess of water. The precipitate is collected preferably by centrifugation, washed free from NaCl, drained, and dried. It may be obtained crystalline by cooling a saturated solution in NaCl solution. This crystalline form affords a valuable means of testing the digestive power of pepsin. Edestin is readily soluble in 1 : 10 NaCl solution, and in dilute solutions of neutral salts. It dissolves readily in dilute HCl.  $\text{HNO}_3$  precipitates it from this solution. To test pepsin, 0.5 Gm. of edestin is dissolved in 100 c.c. of HCl solution 0.25 : 100, and filtered. Twenty c.c. of the filtrate in a test tube is plunged in a water-bath at  $50^\circ\text{C}$ . After 10 minutes 0.02 Gm. of pepsin is added and the liquid is maintained in the water-bath at  $50^\circ\text{C}$ . for exactly 15 minutes. The tube is then plunged in water at  $17^\circ\text{C}$ . and treated at once with 30 drops of  $\text{HNO}_3$  1.139. If the pepsin is of official strength, no precipitate will then occur. At the most, a slight opalescence will be observed, not greater than that afforded by one drop of milk in 20 c.c. of water. If instead of having the official strength, 100, the pepsin is only equivalent to a proteolytic index of 90, the opalescence will equal that of 2 drops of milk in 20 c.c. of water. Pepsin of a standard of 80 gives a distinct turbidity; one of 60, a precipitate; and one of 40, an abundant precipitate by this test. A series of tubes may be used and a time standard established. Thus, pepsin of  $80^\circ$  proteolytic power will require 21 minutes for complete digestion of edestin; one of  $60^\circ$ , 27 minutes. Pepsin of maximum activity will render edestin noncoagulable by  $\text{HNO}_3$  under the conditions of the test, in 15 minutes. The results obtained by this method are definite; the physical condition of

the edestin is constant, and the test may be completed in 30 minutes. The same method cannot be applied to the assay of pancreatin, for edestin is not completely digested by that ferment. (See also *Y.B.*, 1914, 28, 90, 267.)

**Philothon.** J. de Rey Pailhade. (*Comptes rend.*, 1915, 160, 37.) The crystalline lens of animals' eyes contains a peculiar albumin characterized by having a portion of its hydrogen in a loosely combined state. The same albumin has been found in the striated muscular tissue, in the liver, and in the embryos of certain seeds. Since it readily combines with free S at 40°C., with formation of  $H_2S$ , it has been named philothon. Its occurrence in the crystalline lens points to the affinity, in a chemical sense, of this structure with the voluntary muscles. This explains the similar physiological changes which occur in old age in the two tissues; and also explains the curative effect of such salts as KI and  $Na_2CO_3$  in cases of cataract.

**Proteolytic Activity of Pancreas Preparations, Comparison of Methods for Determining.** J. H. Long and A. W. Barton. (*J. Amer. Chem. Soc.*, 1914, 36, 2151.) The proteolytic value of six pancreas preparations has been determined by four distinct methods; the metacasein reaction of Roberts; a modification of the Fuld-Gross reaction with sodium caseinate; the formaldehyde titration of amino-acids liberated in digestion; and the fibrin digestion. It was hoped to find such relations as would permit the translation of activity as expressed on a given standard in terms of another. By these four methods the activities of the six preparations are arranged in the same general order, that is, the strongest ferment by the first method is found to be the strongest by the others. For the weakest preparations the order is about the same. But the relative rank, quantitatively, of the different ferments is very different as measured by the different methods. While the strongest ferment by the metacasein reaction appears to be about 12 times the strength of the weakest, and about 10 times as strong by the digestion of fibrin, by the other tests the relation is as 2 or 3 to 1. Even greater irregularities appear in comparing some of the other ferments.

It is not possible at the present time to translate the proteolytic value of a tryptic ferment from the terms of one standard to the terms of another, with the products as at present furnished by chemical or pharmaceutical dealers, because these preparations are made by very different processes of extraction, concentration

or activation, which leave, probably, mixtures of ferments in widely different proportions in the finished products, and unknown amounts of inorganic salts. There is evidence to suggest that the products sold as trypsins or pancreatins contain at least two different enzymes reacting in different ways with proteins. The effects observed in any case are mixed effects depending on the proportions in which the enzymes are present. These enzymes possess different degrees of thermostability. The desirability of a more rational definition of trypsin is pointed out. The definition should include a statement of the essential points of manufacture and should be authorized by some responsible body such as a pharmacopœial revision committee. Since what is called trypsin is prepared for the use of medical men, these users are entitled to the fullest knowledge concerning the composition and properties of the product. There is no excuse for secrecy here and products should be made to conform to interchangeable standards. The various methods employed are fully described in the text. (See also *Y.B.*, 1909, 163; 1912, 44, 46, 54; 1913, 140; 1914, 26, 28, 89, 266.)

**Serums, Anti-Snake Bite, Preparation of, in Brazil.** J. Boyer. (*Scientific American*, 112, No. 20; *Chem. News*, 1915, 111, 281.) In the Serotherapeutic Institute of Brazil, Butantan, Dr. Vital Brazil produces serums for the cure and prevention of the effects of snake bites. The snakes used in preparing the serums are kept in a small park, containing numerous dome-shaped shelters, which is surrounded by a wall and a ditch filled with water. Other specimens are kept in a similar park, near the main building, in order to study their habits, favourite food, the very diverse venomous properties of various species, and the best method of escaping their attacks. The hot and moist forests of Brazil swarm with venomous serpents, but the slightest noise alarms the peaceful and timid reptiles, which attack only those persons and animals that tread on them or destroy their lairs. The principal families are the Bothrops and the Crotalæ, or rattlesnakes. The Bothrops venom decomposes the blood and produces internal hæmorrhage, with intense congestion of the liver, kidneys, and brain, while the venom of the Crotalæ paralyzes the respiration, circulation, and vision, and usually causes death within twenty-four hours. Each venom requires its special antidote. Dr. Brazil prepares a serum for each, and also a polyvalent or compound serum, which is effective against all

Brazilian snake venoms, for use when the species of the attacking snake is unknown. The serums are obtained from young and sound horses and asses, which receive, at intervals of five or six days, injections of venom, increasing from one-twentieth Mgm. to 1 Gm. A year's treatment is required to produce perfect immunity and an effective serum. The polyvalent serum is obtained by injecting the venoms of Bothrops and Crotales alternately. The animals thus immunized furnish anti-venom serum for a long time, if they receive a fresh injection of venom after each extraction of serum. Tubes of serum, with hypodermic syringes, are sent gratuitously to hospitals, municipalities, and poor patients. Others are sold at low prices or exchanged for live snakes. In 1913 about 900 tubes of rattlesnake serum, 800 of Bothrops serum, and 4,500 of polyvalent serum were distributed, and 4,500 snakes were received. Serums for plague, diphtheria, and tetanus also are produced by the usual methods. In the course of his study of Brazilian serpents, Dr. Vital Brazil has discovered a non-venomous constrictor snake, the *mussurana*, which is naturally immune to snake venom, and which kills venomous snakes by crushing them in its coils and then devours them. It is a remarkable fact that the serums prepared at Lille by Dr. Calmette, the originator of the serum treatment for snake bites, are powerless against the venom of Brazilian serpents.

**Silks, Artificial, Identification of.** L. J. M A T O S. (*Chem. Engineer*, 1914, 20, 209; *Analyst*, 1915, 40, 68.) The following tests are given for distinguishing between the various commercial artificial silks: A small tuft of the silk is heated in a dry tube, and the vapours are tested with litmus-paper. Alkaline vapours indicate gelatin silk, acid vapours silks of cellulose basis. With iodine- $\text{ZnCl}_2$  and iodine- $\text{O}_4$ , the reagents being made at suitable concentration, cellulose acetate silk is stained yellow, and cellulose silks reddish-violet or blue. In cold concentrated  $\text{H}_2\text{SO}_4$  collodion and viscose cellulose silks dissolve rapidly, cuprammonium silks only slowly. In warm 40 per cent. KOH solution cellulose and cellulose acetate silks are swollen, while gelatin silk dissolves rapidly and completely. In cuprammonium reagent cellulose silks swell and dissolve, cellulose acetate silk swells without dissolving, and gelatin silk takes a bluish-violet colour without dissolving. The nickel-oxide-ammonia reagent, both cold and warm, causes swelling of cellulose and cellulose acetate silks without dissolving; gelatin silk takes a brown colour without

dissolving. Alkaline copper glycerol solution has no action on cellulose and cellulose acetate silks on boiling, but gelatine silk dissolves in a short time. Diphenylamine- $\text{H}_2\text{SO}_4$  gives a blue colour with collodion silk, and serves to distinguish this from the other varieties of cellulose silk. Dyed samples should be "stripped" by means of hydrosulphite before applying the reagents.

**Thyroid Gland and Iodine.** F. Blum and R. Grützner. (*Z. physiol. Chem.*, 91, 400, 450; *Chem. Abstr. Amer. Chem. Soc.*, 1914, 8, 3587.) Most of the I of the thyroid gland is present in the form of a compound with protein, but a small portion exists in the form of compounds soluble in acetone; among these the presence of free alkali iodide can be demonstrated. Alkali iodide occurs in this way, even when it cannot have been contained in the food. The quantity of I found in the thyroid varies greatly; in the sheep 1-1.5 Mg. per gland is an average value; in the dog the quantity is less on the average but varies more. When alkali iodide is administered, the amount of I in the thyroid increases, but it is present there in organic combination. The iodo-protein of the thyroid (thyreoglobulin) has not a constant I percentage; this increases after the administration of alkali iodide. Administration of alkali iodide after the removal of 1 lobe of the gland causes an increase in the amount and I percentage of the thyreoglobulin in the remaining lobe. When I is no longer present in the food, it remains in the thyroid, and if it was present there in unusual amount before, the quantity remains high afterwards. I found in the blood can be considered of thyroid origin only if it is in organic combination, and this has not been found. I in inorganic combination when present in the blood has arisen from the food and is of transient occurrence. Animals fed with I-free food have no inorganic I in their blood although their thyroids contain much I. After administration of alkali iodide "inorganic" I can be detected in the blood for a long time. "Organic" I, probably of thyroid origin, can be found in some of the cases under some pathological conditions (eclampsia, nephritis).

**Thyroid Gland, The Human Foetal, Presence of Iodine in.** F. Fenger. (*J. Biol. Chem.*, 1915, 20, 695; *Chem. Abstr.*, 1915, 9, 1636.) Both enlarged and normal human foetal thyroids contain I at least during the last three months of intra-uterine life. Normal sized foetal glands contain relatively more I and

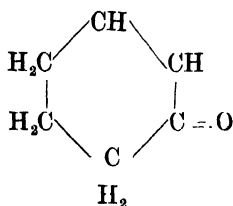
less P than enlarged glands. These conditions are analogous to those existing in the foetal and adult thyroids from cattle, hogs and sheep.

**Thyroid Gland, Seasonal Variation in Composition of.** A. Seidell and F. Fenger. (*Bulletin Hygien. Lab. U.S. Public Health Service ; Pharm. J.*, 1914 [4], 39, 868.) As the result of the analyses of a great number of ox, hog, and sheep thyroids, totalling many thousands, and taken twice in each month throughout the year, the following conclusions are arrived at. The occurrence of a seasonal variation in the I content, and consequently the activity of the thyroid gland, is confirmed by analyses of samples collected during a year. It is furthermore shown that a regular change occurs in the P and ash content of thyroids, and the amounts of these constituents vary inversely with the iodine. This is explained on the assumption that P does not form a part of the active iodine complex of the gland, but only of the supporting glandular tissues. An increase in the percentage of I would, therefore, naturally be accompanied by a decrease of P. Consequently, it appears that neither the composition of the active iodine complex nor that of the supporting tissues of the gland changes with season, but only the relative amounts of the two. In regard to the fresh weights of the glands, the results show a more or less regular seasonal change coincident with the iodine in the case of the ox and sheep, but not with the hog. The results of Martin and Guyer upon the thyroids of English sheep confirm the author's observations in regard to the seasonal change in the activity of the gland. A consideration of the several causes of the seasonal change in activity of the thyroid leads to the conclusion that the temperature factor is the most important of all. The activity of the thyroid diminishes regularly from about September to the following March or April, and then abruptly changes to a regular increase in activity from March or April to the following September. Numerous tables and graphs are given comprising the results of an immense amount of work. (See also *Gen. Index* and *Y.B.*, 1911, 140 ; 1912, 48, 408 ; 1913, 39, 487.)

**Trigonocephalus Blomhoffi, a Poisonous Japanese Snake, Constituents of.** S. Suguro. (*Jap. P.J.*, 1914 [387] ; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 664.) This venomous snake, known as "*Mamushi*," has long been used in medicine, dried, in the form of powder, as a tonic by the Japanese. The Et,O

extract of the drug contains cholesterol and fat. The EtOH extract gives an unknown crystalline N-containing substance, taurine, and much protein.

**Urinod, The Odorous Constituent of Urine.** W. M. Dehn and F. A. Hartman. (*J. Amer. Chem. Soc.*, 1914, **36**, 2136.) The main odorous principle of urine is a neutral, yellow, oily liquid, slightly denser than water, in which it is insoluble, having an intense, persistent penetrating urinous odour. It has been named *urinod* and has the empirical formula  $C_6H_8O$  and may



possibly be a cyclo-hexenone isomer. It is extremely toxic and may have some influence in toxæmia. Urinod is readily oxidized; hence oxidizing agents are best as deodorants for urinals. The odour of the pure substance is very persistent; one drop on filter paper retained its odour for 15 months. It occurs in normal urine in a conjugated state from which it is liberated by mineral acids. It was isolated by acidifying 1,000 litres of urine with  $\text{H}_2\text{SO}_4$ ; distilling; shaking out the distillate with  $\text{Et}_2\text{O}$ ; removing acids, phenols and bases from the  $\text{Et}_2\text{O}$  extract; distilling off the solvent; redistilling the residue in aqueous vapour; again shaking out the distillate with  $\text{Et}_2\text{O}$ ; shaking up the  $\text{Et}_2\text{O}$  extract with Hg to remove S; removing the solvent and fractionating *in vacuo*. Urinod boils at  $108^\circ\text{C}$ . under 28; mm. or  $208^\circ\text{C}$  with decomposition under normal pressure.

**Wool Fat, Detection of Hydrocarbon Oils in.** G. Tellera. (*Boll. Chem. farm.; Pharm. Zentralh.*, 1914, **55**, 414.) One Gm. of the purified wool fat is dissolved in 15 c.c. of pure  $\text{Et}_2\text{O}$  and filtered. To the filtrate, 5 c.c. of absolute EtOH is added. In presence of 2 to 3 per cent. of soft paraffin a precipitate is formed. With 1 per cent. of paraffin the precipitate appears in half an hour. The test is not definite with crude or partly purified wool fat. In this case, the saponification value must also be determined.



## CLINICAL TESTS

**Amoebae, Parasitic, Method for Staining.** A. Marshall. (*Wellcome Laboratory Journ.*, Sept. 1914; *Lancet*, 1915, 188, 145.) Smears are made from suspected stools and transferred rapidly, while still wet, to Schaudinn's fluid. They are then washed in EtOH of different strengths and finally in distilled water, after which they are stained in Delafield's hæmatoxylin for 20 minutes. They are next washed in tap water and stained with carbolfuchsin, as for tubercle bacilli; after which they are again washed with water and finally differentiated with Sprengel's solution of picric acid, consisting of equal parts of absolute EtOH and of saturated watery solution of the acid. This is applied for three to five minutes, during which time the reagent is changed three or four times. The stained films are then dehydrated in absolute EtOH cleared in xylol, and mounted in Canada balsam. Thus treated, the nuclei of the parasites are stained a purplish black, while the cytoplasm is a pale translucent yellow colour. Red blood corpuscles are also stained yellow. The method is easy, rapid, and certain in its results.

**Bacteriology of Gaseous Gangrene.** A. Sartory and L. Spillmann. (*Comptes rend.*, 1915, 160, 210.) The chief organism causing gaseous gangrene is identified with the anaerobic *Bacillus perfringens*. Cultures of this produce typical lesions in guinea-pigs. It is accompanied by other organisms, staphylococci and streptococci, but these are considered to be of minor importance. A lanceolated diplococcus has also been found in some cases, which may ultimately prove to be identical with the organism described Weinberg.

**Cerebro-Spinal Fluid, Normal or Pathological, Simple Reaction to Differentiate.** P. Boveri (*Muench. med. Woch.*, *B.M.J. Epit.*, 1915, 1, 16) finds the following reaction useful to distinguish between normal and pathological cerebro-spinal fluid. In a small test tube, containing 1 c.c. of cerebro-spinal fluid, 1 c.c. of a 0.01 per cent. solution of  $\text{KMnO}_4$  is added so that it slowly trickles down the side of the tube, which is held slanting. When the tube is now held upright, a more or less bright yellow colour is observed at the junction of the two fluids, provided the cerebro-spinal fluid is pathological. If it is normal, there is no coloration. On shaking the two fluids so as to mix them, if the cerebro-spinal

fluid is normal, the rosy-violet colour of the mixed fluids lasts for a considerable time. But if the fluid is pathological, this colour changes to bright yellow in a few seconds to a few minutes. This reaction is strong when it occurs within two minutes, it is of medium strength when it occurs in three to four minutes, and it is weak when it requires five to six minutes. When this time is exceeded, the reaction may be regarded as negative. The reaction is definite evidence of pathological changes in the cerebrospinal fluid, and is more delicate than those hitherto in use, including Nonne's and Noguchi's.

**Diphtheria Bacillus, Ponder's Modified Stain for.** J. J. Kenyon. (*Am. J. Pub. Health*, 1915, 5, 246; *Chem. Abst.*, 1915, 9, 1339.) Toluidine blue, 0.1 Gm., azure 1, 0.01 Gm., methylene blue, 0.01 Gm., glacial AcOH, 1 c.c., H<sub>2</sub>O 120 c.c. and 95 per cent. EtOH, 5 c.c. Preparations are made in the usual way, fixed with heat, cooled and stained 2 minutes or more (no overstain at 7 minutes). The bacilli stand out with granules clear, sharp and of dark red colour, while the rod is stained a pale blue. It is valuable in release cultures and in excluding Hoffman's bacillus, which has no granules.

**Faeces, Determination of Fat in.** F. C. Gephart and F. A. Csonka. (*J. Biol. Chem.*, 1914, 19, 521-31; *Chem. Abst. Amer. Chem. Soc.*, 1915, 9, 329.) From 2-3 Gm. of dried faeces, or 5-7 Gm. moist faeces, is weighed into a cylindrical flask of 175 c.c. capacity. Add 20 c.c. 95 per cent. EtOH, 4 Gm. KOH and a glass bead. Boil under reflux for 1 hour. Cool, add 50 c.c. H<sub>2</sub>O, 20 c.c. 20 per cent. HCl, in 4 portions, cooling thoroughly. Add 50 c.c. Et<sub>2</sub>O and shake well. Rinse off stopper with Et<sub>2</sub>O and insert another, with tubes like a wash bottle, and blow off the layer of Et<sub>2</sub>O into a 250 c.c. separatory funnel. The extraction is repeated twice with 50 c.c. Et<sub>2</sub>O and once with 25 c.c. The combined Et<sub>2</sub>O extracts are washed four times with 50 c.c. H<sub>2</sub>O. Transfer the Et<sub>2</sub>O to a 300 c.c. Erlenmeyer and evaporate the Et<sub>2</sub>O, finally drying *in vacuo* over P<sub>2</sub>O<sub>5</sub> overnight. Add 50 c.c. of petroleum ether (b. p. 40-60°), let stand 1 hour, heat to boiling and titrate with N/10 KOH in EtOH, 1 c.c. of which is equivalent to 0.0297 Gm. tristearin. (See also *Y.B.*, 1912, 51; 1913, 42, 43.)

**Sodium Tellurite as a Rapid Test for the Viability of Tubercle Bacilli.** H. J. Corper. (*J. Infect. Dis.*, 1915, 16, 47-53;

*Chem. Abstr.*, 1915, 9, 1340.) In bactericidal experiments in connection with chemo-therapeutic work the author attempted to use the Gosio vital reaction ( $\text{Na}_2\text{TeO}_3$ ) as an index of life in virulent human tubercle bacilli; he did not find it an available general reagent for this purpose, at least by the methods tested. He was, however, able to develop with its use a simple rapid test for determining the viability of cultures of tubercle bacilli of value especially in eliminating such loss of time as may be occasioned by working with dead instead of viable cultures. A small clump of the culture to be tested is placed in the cup of a sterile, hollow glass slide, and 1 or 2 drops of sterile 0.2 per cent.  $\text{Na}_2\text{TeO}_3$  in distilled water are added; it is then covered with a sterile cover slip bordered with sterile vaseline, and placed in the incubator at  $37^\circ\text{C}$ . Life of the organism is indicated by the blackening of the lump of culture, which occurs in from 30 minutes to 2 hours.  $\text{Na}_2\text{TeO}_3$  is lethal for rabbits when given intravenously in amounts of about 0.8 Mgm. per kilo. It does not kill tubercle bacilli even at a concentration of 0.01 per cent. in  $\text{NaCl}$  solution or glycerol broth for 48 hours at  $37^\circ$ , nor does it inhibit the growth in 0.001 per cent. concentration on glycerol agar.

**Sputum, The Albumin Reaction in.** C. Artom. (*Riv. osped.*, 3, 256-60; *Chem. Abstr.*, 1914, 8, 2901.) The albumin reaction is always positive in tuberculosis; it is more intense when the number of bacilli is high, but is quite distinct when no bacilli are detected. The albumin reaction is not specific for tuberculosis; it is almost always positive in heart disease, nephritis, pneumonia, and broncho-pneumonia, where it disappears with healing; always negative in acute and chronic bronchitis and in emphysema without complications. The albumin reaction is connected with the formation of an exudate or transudate. (See also *Y.B.*, 1911, 50; 1912, 57; 1913, 46.)

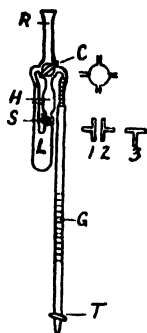
**Syphilis, New Reaction for Diagnosis of.** W. Landau. (*Presse méd.*; *Schweiz. Apoth. Zeit.*, 1915, 53, 50.) The reagent employed consists of 1 : 100 solution of I in methane tetrachloride. Two volumes (conveniently 0.2 c.c.) of the serum is treated with 1 volume of the reagent, without shaking. In 4 hours, at the normal temperature syphilitic serum is clear, transparent and yellow. Normal serum will be opalescent and of a greyish white colour. The test is claimed to be more sensitive than that of Wassermann.

**Tetanus, Presence of Sugar and Urea in Cerebro-Spinal Fluid in Cases of.** O. Ferrier. (*J. Pharm. Chim.*, 1915, 11, 21.) Excess of sugar and of urea was noted in every one of six specimens of cerebro-spinal fluid, drawn from cases of traumatic tetanus, before injection with anti-tetanic serum. All the fluids were absolutely sterile, free from micro-organisms and pathological elements. The amount of glucose, however, from 0.700 to 0.900 Gm. per litre was about double the normal figure. The urea, from 1.000 to 1.350 Gm. per litre indicated a state of grave uraemia.

**Typhoid Bacteria, Rapid Isolation of, by Means of a Sand Filter.** P. Carnot and B. Weill-Hallé. (*Comptes rend.*, 1915, 160, 148.) The following method is recommended for the rapid and certain isolation of the typhoid bacillus and is specially serviceable for tracing typhoid "carriers." It depends on the well-known greater mobility of the *Bacillus typhosus*, which enables it to penetrate a sand filter more quickly than the organisms which accompany it. A glass tube somewhat over 33 cm. long and 5 to 6 mm. internal diameter is slightly drawn out in the middle, then bent in U form. Pure sterile sand is introduced into the constricted portion so that the total length of the sand column is about 10 cm. Sterile bouillon or other liquid culture medium is then introduced into one branch and allowed to filter through the sand until a column of liquid about 10 cm. high is over each sand layer. The free ends of the tube are plugged with cotton and the whole is sterilized. An evacuant enema is administered to the suspected subject, followed by an intestinal washing which affords an almost clear liquid. A few drops of this is mixed with the nutrient broth in one of the arms of the U tube. The whole is then incubated at 37°C. for 18 hours. In the majority of cases of typhoid, the nutrient medium on the other side of the sand layer will be turbid by this time and the specific bacillus may be detected by direct micro-examination and subsequent reactions. Occasionally a second incubation and filtration through sand is necessary to obtain pure cultures of typhoid when the development of the first culture is slow. The method is specially recommended for examining suspected stools. The whole process can be completed in 24 hours, and possibly the time may be still further shortened by decreasing the length of the column of sand.

**Ureometer, Improved.** G. Rodillon. (*Bull. Sci. pharm.*,

col., 1914, 21, 395.) The apparatus consists of a funnel tube (*R*), a 3-way cock (*C*), a bulb for hypobromite solution (*H*), a reaction bulb (*L*) connected to *H* by a stopcock (*S*), and a gasometer tube (*G*) (content 37 c.c.) graduated throughout most of its length in 0.1 c.c. but drawn to a capillary at the top and more accurately graduated and with a stopcock (*T*) at its lower end. In operating,



ing, *S* and *T* are opened, *C* placed at position 1, the urine introduced and washed in with water, then *S* is closed, *C* turned to position 2 and the hypobromite solution introduced into *H*. Then *C* is turned to position 3 and the apparatus placed in a cylinder containing water at room temperature so that the level of the water reaches the centre of *C*. The apparatus is allowed to reach the temperature of the water, then *C* is turned to position 2 till the water reaches 0 in the gasometer tube, then turned back to position 3. *T* is then closed, *S* opened and the apparatus vigorously shaken. It is then returned to the cylinder, *T* opened, and after the apparatus has attained room temperature the level of the water in the gasometer is adjusted to that in the cylinder and the volume of gas read off. (See also Y.B., 1914, 39.)

**Urine, Colorimetric Determination of Uric Acid in.** S. R. Benedict and E. H. Hitchcock. (*J. Biol. Chem.*, 1915, 20, 619; *J.S.C.I.*, 1915, 34, 633.) A modification of a method proposed by Folin and Denis is recommended. From 2 to 4 c.c. of the urine is treated with 5 c.c. of water and about 20 drops of a solution composed of 3 per cent. silver lactate solution, 70, magna mixture, 30, and concentrated AmOH, 100 c.c. The mixture is centrifugated, the liquid then decanted as completely as possible, the sediment is treated with 2 drops of a 5 per cent. KCN solution, 1 c.c. of water, and 2 c.c. of uric acid reagent, 10 c.c. of a 20 per cent.  $\text{Na}_2\text{CO}_3$  solution is added, and, after about 30 seconds, the mixture is diluted to 50 c.c. and the coloration obtained compared with that produced under similar conditions from 5 c.c. of standard uric acid solution. The latter solution is prepared by dissolving 9 Gm. of  $\text{Na}_2\text{HPO}_4$  and 1 Gm.  $\text{NaH}_2\text{PO}_4$  in 300 c.c. of hot water, filtering the solution if necessary, adding 200 Mgm. of pure uric acid and 1.4 c.c. of glacial acetic acid, and diluting the whole to 1 litre; 5 c.c. of  $\text{CHCl}_3$  may be added to prevent the growth of bacteria and moulds. The uric acid reagent is prepared by boiling together 100 Gm. of sodium

tungstate, 80 c.c. of 85 per cent. phosphoric acid, and 750 c.c. of water for an hour and a half under a reflux condenser, and diluting the cooled solution to 1 litre. (See also *Gen. Index* and *Y.B.*, 1904, 525; 1906, 79; 1907, 164, 167; 1912, 58, 62; 1913, 53, 55, 58, 59.)

**Urine, Detection of Sugar in.** W. Cramer. (*Biochem. J.*; *Lancet*, 1915, 188, 1192.) The reagent is prepared by dissolving 0.4 gr. of  $\text{HgO}$ , red or yellow, with 6 gr. of  $\text{KI}$  in 100 c.c. of water, and adjusting the alkalinity of the solution so that 10 c.c. are neutralized to phenol-phthalein by 2.5 c.c. of  $\text{N}/10$  acid. The resulting solution is colourless in the cold and turns slightly yellow on boiling. To test for sugar in urine 3 c.c. of the reagent is heated to boiling; then 0.3 c.c. of urine is added and the mixture again boiled. On removing the test-tube from the flame, the mixture darkens if sugar be present, and a deposit of black metallic  $\text{Hg}$  gradually settles to the bottom. Not only glucose, but lactose, maltose, xylose, and arabinose give the reaction, while cane sugar does not. The sensitiveness of the test fluid can be varied by increasing or diminishing its degree of alkalinity, and the point fixed, as stated above, corresponds to a degree which gives a faint reaction with normal urine, which is known to contain about 0.1 per cent. of sugar. If the reagent is made more alkaline and thus more sensitive, it may give a precipitate with creatinine and other organic substances. The author finds that the "2.5 standard" solution gives a definite reaction with small amounts of sugar in the urine, which only produce doubtful results when tested with Fehling's reagent. He suggests as an indication of the degree of the reaction the possibility of reading print through the solution when boiled in an ordinary test-tube, 30 seconds after this is removed from the flame, a few drops of acetic acid being first added to remove any possible turbidity due to phosphates. Small quantities of sugar can thus be recognized and roughly estimated. Ammoniacal urine must not be used for the mercuric test any more than in performing Fehling's reaction. Possibly for ordinary clinical testing the "2.0 standard" solution, which gives no reaction with normal urine, may seem preferable, but there is no reason to ignore the minute amount of sugar normally present, when small variations from this percentage can be readily demonstrated. (See also *Gen. Index*, and *Y.B.*, 1904, 178; 1907, 166, 171; 1909, 92; 1910, 47, 52, 53; 1911, 52, 1912, 59, 63, 64; 1913, 235; 1914, 36.)

**Urine, Determination of Benzoic Acid in.** G. W. Raiziss and H. Dubin. (*J. Biol. Chem.*, 1915, 20, 125.) One hundred c.c. of fresh urine is acidified with 1 c.c. of  $\text{HNO}_3$ , then saturated with  $\text{Am}_2\text{SO}_4$  (from 50 to 60 Gm. is required), and extracted with 50, 40, 30, and 30 c.c. of pure toluene. The combined toluene extracts are washed twice with 100 c.c. of saturated  $\text{NaCl}$  solution containing 0.05 per cent. of strong  $\text{HCl}$ , and the benzoic acid in the toluene solution is titrated with  $\text{N}/20$  sodium ethylate, using phenolphthalein as indicator. The sodium ethylate is prepared by dissolving 2.3 Gm. of metallic  $\text{Na}$  in 2 litres of absolute  $\text{EtOH}$ . Hippuric acid, if present, is not extracted by toluene.

**Urine, Dimethylaminobenzaldehyde as a Reagent for Bile Pigments in.** Travassô. (*Arch. méd. Angers; Chem. Abstr.*, 1915, 9, 1342.) The reagent consists of 2 Gm. dimethylaminobenzaldehyde, 50 Gm.  $\text{HCl}$ , 50 Gm.  $\text{H}_2\text{O}$ . Four c.c. of the reagent is introduced by pipette into a test tube containing 10 c.c. of the urine to be examined. In case the urine contains bile pigments, a green coloration is instantly produced at the zone of contact, and this colour soon extends to the lower layer of liquid. After a few minutes the two layers become diversely coloured, the lower one being green and the upper one assuming a mahogany coloration peculiar to jaundiced urine. The results obtained with this reagent are in accord with those given by the Gmelin and Grimbert reactions.

**Urine, Quantitative Determination of Albumin in.** O. Folin and W. Denis. (*J. Biol. Chem.*, 18, 273-6; *Chem. Abstr.* 1914, 8, 3312.) *Turbidity method*: To about 75 c.c. water in each of two 100 c.c. flasks is added 5 c.c. 25 per cent. thiosalicylic acid. To one flask is added 5 c.c. standard protein solution containing 10 mg. of albumin and to the other the albuminous urine, 1 c.c. at a time, from an Ostwald pipette until the turbidity approximates that of the standard. The two flasks are filled to the mark and mixed by a few careful inversions (not by shaking, which tends to agglutinate the precipitate). The comparison is then made in the Duboscq colorimeter, the standard being invariably first read against itself. The standard is set at 20 mm. and the unknown must read between 10-30 mm. Dividing 200 by the product of the reading of the unknown and the number of c.c. of urine taken gives the albumin in Mgm. per c.c. of urine. The standard protein solution is prepared from fresh blood serum (either slaughter house or normal

human blood may be used) free from haemoglobin by diluting 25–35 c.c. of serum to 1500 c.c. with 15 per cent. pure NaCl solution, mixing and filtering. The protein content is determined by N determinations and the solution is diluted with 15 per cent. NaCl so as to contain 2 Mgm. of protein per c.c. It is saturated with  $\text{CHCl}_3$  (20 c.c.) and preserved in a refrigerator. The method is not applicable to urines deeply coloured with blood or bile pigments. It is useful in determining the albumin content of such fluids as exudates, transudates and cerebro-spinal fluid. *Gravimetric method*: Ten c.c. urine is pipetted into a weighed conical centrifuge tube and 1 c.c. 5 per cent.  $\text{AcOH}$  is added. The tube is kept in boiling water for 15 minutes and then centrifuged for a few minutes. The supernatant liquid is poured off, the precipitate stirred up with 10 c.c. of boiling 0.5 per cent.  $\text{AcOH}$  and again centrifuged. The liquid is again poured off and the precipitate in the tube is washed with 50 per cent.  $\text{EtOH}$ . After centrifuging and pouring off the supernatant liquid for a third time, the tube is placed for 2 hours in an air bath at  $100^\circ$ – $110^\circ\text{C}$ ., cooled in a desiccator and weighed. (See also *Gen. Index*, and *Y.B.*, 1908, 15; 1910, 51; 1912, 62; 1913, 53.)

**Urine, Determination of Sugar in, by Bang's Method.** W. Gray. (*J. Amer. Pharm. Assoc.*, 1915, 4, 740.) *Preparation of Bang's Solution*.—First make a concentrated solution of chemically pure  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$  in distilled water, using 250 Gm. in 1500 c.c. of water. The amount of Cu present should be determined by electrolysis; a calculation should be made then as to the number of c.c. of solution containing 25 Gm. of  $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ . The number of c.c. may be more or less than 150, according to the  $\text{CuSO}_4$  employed. This may be, for example, that it takes 154.2 c.c. instead of 150 c.c.

*Solution No. 1*.—Weigh accurately into a beaker 500 Gm. of  $\text{K}_2\text{CO}_3$ , 400 Gm. of  $\text{KCNS}$ , 100 Gm. of  $\text{KHCO}_3$  and dissolve in 1200 c.c. of distilled water at  $60^\circ\text{C}$ . The salts should be chemically pure. When dissolved, place in a 2 litre volumetric flask, reduce temperature to  $30^\circ\text{C}$ . and run in, slowly, by means of a burette, 154.2 c.c. of the above concentrated  $\text{CuSO}_4$ , shaking the mixture while this is being added. This is very necessary to prevent precipitation. Finally add enough distilled water to make 2 litres.

*Solution No. 2*.—Dissolve 200 Gm. of  $\text{KCNS}$  and 6.55 Gm. of hydroxylamine sulphate in distilled water at ordinary temperature to make 2 litres of solution.

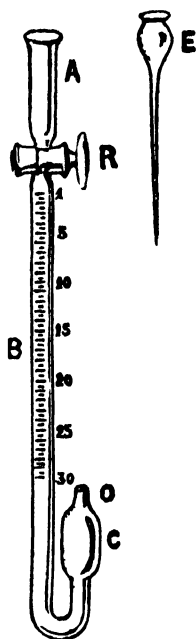


*The Application of the Test.*—1. Measure accurately 10 c.c. of urine into a 200 c.c. flask. 2. Add to the urine exactly 50 c.c. of solution No. 1. 3. Heat this flask on a wire gauze over a Bunsen flame so regulated that the flame turns a small spot of the gauze red. Protect flame from air currents. 4. After boiling commences, allow to boil for exactly three minutes. *Caution.*—If the urine contains more than 0.6 per cent. of sugar, the blue colour will be entirely destroyed. If the blue colour turns yellowish on boiling, a smaller amount of urine must be used. Take 2 or 5 c.c. of urine diluted with water to 10 c.c. and repeat the operations this far. 5. Cool flask and contents to room temperature, quickly, by immersing the flask in cold water. 6. Titrate the contents of the flask with No. 2 solution until the blue colour is exactly decolorized. This titration should be so conducted that the solution runs from the burette rapidly, but in drops. 7. From the number of c.c. of No. 2 solution used, calculate, from the appended table, the sugar in milligrams in the amount of urine used.

BANG'S TABLE OF REDUCTION EQUIVALENTS.

C.c. Hydroxyl- amine Solution used.	Mgm. Sugar repre- sented.	C c Hydroxyl- amine Solution used.	Mgm. Sugar repre- sented.	C c. Hydroxyl- amine Solution used.	Mgm. Sugar repre- sented.	C c Hydroxyl- amine Solution used.	Mgm. Sugar repre- sented.
0.75	60.0	13.00	39.0	25.50	23.5	38.00	10.4
1.00	59.4	13.50	38.3	26.00	22.9	38.50	9.9
1.50	58.4	14.00	37.7	26.50	22.3	39.00	9.4
2.00	57.3	14.50	37.1	27.00	21.8	39.50	9.0
2.50	56.2	15.00	36.4	27.50	21.2	40.00	8.5
3.00	55.0	15.50	35.8	28.00	20.7	40.50	8.1
3.50	54.3	16.00	35.1	28.50	20.1	41.00	7.6
4.00	53.4	16.50	34.5	29.00	19.6	41.50	7.2
4.50	52.6	17.00	33.9	29.50	19.1	42.00	6.7
5.00	51.6	17.50	33.3	30.00	18.6	42.50	6.3
5.50	50.7	18.00	32.6	30.50	18.0	43.00	5.8
6.00	49.8	18.50	32.0	31.00	17.5	43.50	5.4
6.50	48.9	19.00	31.4	31.50	17.0	44.00	4.9
7.00	48.0	19.50	30.8	32.00	16.5	44.50	4.5
7.50	47.2	20.00	30.2	32.50	15.9	45.00	4.1
8.00	46.3	20.50	29.6	33.00	15.4	45.50	3.7
8.50	45.5	21.00	29.0	33.50	14.9	46.00	3.3
9.00	44.7	21.50	28.3	34.00	14.4	46.50	2.9
9.50	44.0	22.00	27.7	34.50	13.9	47.00	2.5
10.00	43.3	22.50	27.1	35.00	13.4	47.50	2.1
10.50	42.5	23.00	26.5	35.50	12.9	48.00	1.7
11.00	41.8	23.50	25.8	36.00	12.4	48.50	1.3
11.50	41.1	24.00	25.2	36.50	11.9	49.00	0.9
12.00	40.4	24.50	24.6	37.00	11.4	49.50	0.5
12.50	39.7	25.00	24.1	37.50	10.9	50.00	0.0

**Urine, Determination of Urea in.** A. Desgrez and R. Moog. (*Comptes rend.*, 1914, 159, 250.) The authors advocate the use of the special form of ureometer figured, using  $\text{CHCl}_3$  to fill it. They find that the addition of a fine, insoluble powder, such as kieselguhr, to the reaction mixture acts as a catalyser and facilitates the rapid and complete decomposition of urea at  $17^\circ\text{C}$ . The reagent employed consists of  $\text{Hg}$ , 50 Gm.  $\text{HNO}_3$ , sp.g. 1.334, 150 Gm. The  $\text{Hg}$  is dissolved in the acid in the cold. One Gm. of kieselguhr is shaken up with 100 c.c of water and allowed to stand for 1 minute. The supernatant liquid, mixed with the  $\text{HgNO}_3$  solution in equal volumes, forms the reagent. The apparatus can be used in any vessel of water. It is composed of a tube *B* graduated in 0.1 or 0.05 c.c. bearing the cup *A*, from which it is separated by the perforated stopcock *R*. It is bent in a U at the lower extremity to form the short ascending limb, which bears the bulb *C*. The aperture *O* is closed with the finger, and  $\text{CHCl}_3$  is introduced into the tube *B*, by means of the fine beaked funnel *E*, the drawn-out beak of which will pass through the aperture of the stopcock *R*. When the level of the  $\text{CHCl}_3$  reaches the middle of the reservoir *A*, the finger is withdrawn from *O* and *B* is filled therewith. *R* is then closed. The apparatus is clamped in a stand, alongside the vessel of water. The measured volume 1 c.c. of urine is then run in, followed by a washing of water, 2 c.c., and then by the reagent, 6 c.c. At first reaction is active. The apparatus is stood in the vessel of water at about  $25^\circ\text{C}$ . for 30 minutes when it is withdrawn. The orifice *O* is closed and the mixture in the tube agitated by turning over, but avoiding emulsifying the  $\text{CHCl}_3$ . This is done twice or thrice at 2 minutes interval. The ureometer is then fixed and the gas washed by passing about 150 c.c. of water through the reservoir *A*, with thorough agitation. The greater part of the  $\text{CO}_2$  is thus removed. The rest is taken up by the addition of 2 c.c. of equal volumes of  $\text{KOH}$  solution and water, which is run in slowly. The  $\text{CHCl}_3$  is then adjusted to the same level in both limbs by inclining the apparatus, keeping the shorter limb above



the longer one. This effected, the apparatus is stood again in the water and the reading taken.

**Urine, Determination of Urea in, and Indirectly of Allantoin, by Means of Urease.** R. H. A. Plimmer and R. F. Skelton. (*Biochem. J.*, 1914, 8, 70; *Analyst*, 1915, 40, 61.) The determination of urea in urine may be quickly and accurately made by decomposing it with urease (1 Gm. powdered soya-bean) at 35° to 40°C. for one hour. By fitting together three or four cylinders and Allihn bottles in series with a  $\text{H}_2\text{SO}_4$  bottle at the end, duplicate estimations of ammonia and urea in urine can be carried out simultaneously. In the cylinders for the urea estimations are placed 50 to 60 c.c. of water, 1 Gm. of finely ground soya-bean, and 5 or 10 c.c. of urine. These cylinders are kept in a water-bath at a temperature of 35° to 40° C., an air current being drawn through the series. After about an hour the cylinders and bottles are disconnected, and 1 Gm. of anhydrous  $\text{Na}_2\text{CO}_3$  is dropped into the cylinders; they are then connected together again, and the air current drawn through for another hour. To prevent frothing liquid paraffin B.P. is used; it is superior to petroleum or toluene, as it does not evaporate, and it obviates the necessity of using a tube containing cotton-wool between the cylinder and Allihn bottle. The Allihn bottles are charged with excess of N/10  $\text{H}_2\text{SO}_4$  (25 to 50 c.c.), which is titrated with N/10 alkali, using alizarin red as indicator. The method was tested on solutions containing different amounts of pure urea, and the results were found to agree with a N determination by Kjeldahl's method. Urease does not decompose allantoin; and since both allantoin and urea are quantitatively decomposed by the  $\text{MgCl}_2$  method of Folin, the amount of allantoin in those urines which contain both compounds is readily estimated by difference.

**Urine, Simple Method of Determining Glucose in.** A. F. Dimmock. (*B.M.J.*, 1914, 2, 399.) Ten c.c. of urine is diluted to 200 with distilled water. A solution of  $\text{K}_2\text{CO}_3$ , 2 oz. to 6 oz. of distilled water, is filtered and made up to 8 oz. To 20 c.c. of the diluted urine 10 c.c. of the  $\text{K}_2\text{CO}_3$  solution is added in a small flask, and this is boiled carefully for three minutes, and when cool made up to a definite amount, say 50 or 100 c.c., with distilled water. In order to estimate the amount of sugar present, a solution of pure glucose is prepared, 1 Gm. in 200 c.c.

of distilled water ; 20 c.c. of this and 10 c.c. of the  $K_2CO_3$  solution are boiled together in a small flask for three minutes, and when cool made up to 50 c.c. or 100 c.c. The two solutions are then compared by holding the glass tubes over a piece of white paper at an angle of  $45^\circ$ . By pouring the liquid from the known solution into a measure glass until the tints of both are alike, and observing the amount of the known glucose solution used, the percentage can be readily determined ; for example, if 27 c.c. of the pure glucose solution were required for the solution, then, multiplying by two, 54 is obtained as the percentage of glucose in the urine. Albumin, if present, should be removed by acetic acid and heat. The colouring matters in urine do not seem to have any effect, but these could easily be removed by lead acetate or subacetate, taking care not to use excess, filtering or allowing to stand ; any slight excess of lead is easily soluble in the  $K_2CO_3$  solution. The colouring matter of rhubarb and senna becomes reddish-brown with alkali before heating, and samples containing catechol acquire a brown colour on exposure to air.

**Urine, Volatile Substances in.** W. M. Dohn and F. A. Hartmann. (*J. Amer. Chem. Soc.*, 1914, **36**, 2118.) A review of hitherto known volatile substances of urine shows that they are not responsible for its characteristic odour. Some of these, as ammonia, indole and possibly the phenols, contribute to the composite nature of the odour. At ordinary temperatures the volatile acidity of urine is very minute. Direct extraction of urine with  $Et_2O$  yields only a little volatile substance. Distillation of urines with dilute  $H_2SO_4$  yields the largest quantities of volatile substances. These distillates were extracted with  $Et_2O$ , concentrated and distilled. The volatile substances were separated into four major fractions : (a) acids, (b) phenols, (c) bases, and (d) neutral substances. The four fractions distil over a wide range of temperatures, hence they are, respectively, mixtures of many substances. The principal volatile acid was found to be benzoic acid (formed by hydrolysis of hippuric acid),  $H_2S$ , the fatty acids up to heptylic acid, and possibly hexahydrobenzoic acid. The principal phenols are phenol and *p*-cresol ; other higher phenols occur in notable quantities. Methylamine and indole occur as a trace in fresh urines and in larger quantities in fermented urines. The neutral substances of urine are the most important contributors to its odour. (See p. 37.)

**Urine, Simple Test for Acetone in.** F. E. Niece. (*Drugg. Circ.*, 1914, 58, 711.) A reagent is prepared with ammonium nitrate, 30 Gm.; sodium nitroprusside, 2 Gm.; distilled water, enough to make 80 c.c. Mix in the order given, dissolve and keep in well corked vessels away from direct sunlight. To 5 c.c. of urine in a test tube add 0.5 to 1.0 c.c. of the above reagent, mix, then carefully add by contact 3 c.c. of a 10 per cent. solution of AmOH. If acetone is present, there is immediately produced at the zone of contact a purple or "burgundy red" colour. The colour diffuses mostly upwards into the AmOH stratum on standing, and its depth of colour is in proportion to the amount of acetone present. With small amounts of acetone the colour is at first a deep rose pink, soon changing to a purple or violet. After 15 to 20 minutes the colour slowly fades, so that within one hour it is almost discharged. The following confirmatory tests are of interest: If about 3 to 4 c.c. of the highly coloured solution is carefully warmed in a test tube until the purple colour is discharged, a yellow-coloured solution remains; the original colour can be slowly restored by cooling, adding an equal amount of water and vigorously agitating. On standing for about five minutes the colour produced in this manner cannot be distinguished from the one originally produced, unless close comparison be made with a control. If one part of the solution in which a fairly strong colour reaction has taken place is added to 40 or 50 parts of water, the "burgundy" colour will still be plainly perceptible. Even in a dilution of 1 in 200 the colour is plainly seen in a Nessler tube. Five c.c. of a 1 in 800 dilution of commercial acetone in water gave a very distinct reaction on standing for about 15 minutes. The above formula makes a solution that keeps indefinitely, and is not interfered with by excessive amounts of glucose, albumin, indican or creatinin, as is liable to take place with other modifications. The action of the ammonium salt in the test is that of a stabilizer and catalyzer. (See also *Gen. Index*, and *Y.B.*, 1904, 177; 1906, 78; 1908, 204; 1910, 51; 1911, 51, 1913, 54.)

## COLOURING MATTERS

**Abrus Precatorius, Red Colour of Testa.** G. L. Sarkar. (*Biochem. J.*, 1914, 8, 281; *Chem. Abst.*, 1914, 8, 3809.) The colouring matter was precipitated from the aqueous extract of the red seed coats as a Cu compound. This was dissolved

in HCl, the Cu precipitated as CuS and the aqueous portion evaporated to dryness. This residue is stated to give the colouring matter on extraction with water. It is a tannin substance.

**Aniline Colours, Interference of, with Alkaloidal Assays.** G. E. F'Ve and C. E. Vanderkleed. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1681.) Alkaloidal preparations have been met with on the American market, such as powdered extract of stramonium, which are coloured green with an aniline dye to simulate the chlorophyll of the leaf. In these the colouring matter appears in the alkaloidal residue, and being basic vitiates the final titration. In one specimen the colour present increased the alkaloidal value by as much as 24 per cent. The interfering colour can generally be eliminated by shaking out with  $\text{CHCl}_3$ , in the presence of free acid, before liberating the bases with alkali.

**Aniline Dyes in Wine, Detection of.** F. Wohack. (*Chem. Zentr.*, 1914, 1, 1976.) A sample of wine is extracted with amyl alcohol and the amyl alcohol extract warmed on the water-bath with a woollen thread. In presence of an aniline dye the wool is coloured pink and the colour is not changed to green on the addition of  $\text{AmOH}$ . The wine is shaken with  $\text{HgO}$  to remove natural colouring matters, filtered, and made slightly acid with HCl, any precipitated  $\text{HgCl}$  being filtered out. The filtrate heated with a few woollen threads will dye these if aniline colours be present.

**Anthocyan.** R. Wilstaetter and pupils. (*Annalen*, 1915, 408, 1, 15, 42, 61, 83, 110, 122, 136, 147.) *Colouring matter of the rose.* With T. J. Nolan. The chief anthocyan of *Rosa gallica* petals is cyanin, the colour of the cornflower. This flower also illustrates the many varieties of colour, depending upon the acid, neutral or alkaline reaction of the cell sap. The white and yellow rose contain no anthocyan, the rose-coloured a little, while the dark red varieties contain comparatively large amounts. The dry meal of rose petals, when covered with  $\text{MeOH}$ , becomes violet and expands. It yields a violet extract, which soon becomes colourless. It behaves in the same way towards  $\text{H}_2\text{O}$ . With  $\text{EtOH}$  the decolorization is slower. It proceeds very quickly if extracted with a mixture of 4 parts  $\text{MeOH}$  and 1 part  $\text{AcOH}$ . The most suitable solvent

is MeOH containing 2 per cent. HCl. This dark violet-red solution shows the peculiar behaviour of turning darker upon standing, the intensity being doubled in two days. This is not due to the pseudo base of cyanin, since this is changed into the dye with great difficulty in MeOH-HCl, nor to the oxidation of a leuco-compound, for passing air through the solution does not increase the rate of colour increase. It may be due to an unknown anthocyan, whose colourless form is slowly changed into the dye under these conditions. Cyanin is isolated from the rose by extracting 1 kg. of dry powder with 3 litres of 2 per cent. MeOH-HCl for 16 hours, filtering, washing several times with 1 per cent. MeOH-HCl and then extracting with 2 litres of MeOH-HCl and washing with 1.5 litre of 1 per cent MeOH-HCl. The combined filtrates, 5 litres, are precipitated with 2.5 vols. Et<sub>2</sub>O, giving a dark, hygroscopic, resinous precipitate, which is rubbed, while still moist, with 200 c.c. MeOH, 140 c.c. glacial AcOH added and the mixture allowed to stand 24 hours. This yields the pure cyanin as a microcrystalline precipitate.

*Colouring matter of the whortleberry.* With H. Mallison. The method of extracting the colour from the fresh skins is given. The pseudo-base *idacín*, C<sub>21</sub>H<sub>21</sub>O<sub>11</sub>, forms salts which are described.

*Colouring matter of Scarlet Pelargoniums.* With E. K. Bolton. The method of extraction, and the salts, compounds and hydrolysis compounds formed by *pelargonin*, C<sub>27</sub>H<sub>20</sub>O<sub>16</sub>, are described. The chloride, C<sub>27</sub>H<sub>21</sub>O<sub>16</sub>Cl, forms scarlet hair-like needles with a greenish lustre.

*Colouring matter of the larkspur.* With W. Mieg. *Delphinin*, C<sub>41</sub>H<sub>38</sub>O<sub>21</sub>, is described, with its method of extraction, salts, compounds and hydrolysis products. Delphinin chloride, C<sub>41</sub>H<sub>38</sub>O<sub>21</sub>Cl·2H<sub>2</sub>O, forms dark prismatic reddish-brown tables.

*Colouring matter of the grape and bilberry.* With H. Zollinger. Grape skins, freed from pulp, gave *enin*, the chloride of which, C<sub>23</sub>H<sub>25</sub>O<sub>12</sub>Cl, was obtained in dark red prisms with a beetle-green lustre. Bilberry skins yielded *myrtillin*, the chloride of which forms dark red-brown flat prisms with a coppery lustre.

*Colouring matter of Althea rosea.* With K. Martin. The chloride of *althein* is also obtained in large prisms with a bronze lustre.

*Colouring matter of wild mallow.* With W. Mieg. *Malvin*, the colour of *Malva sylvestris* flowers, was isolated as picrate. This was converted into chloride forming dark brownish-red prisms or needles from hot dilute HCl.

*Colouring matter of peony.* With J. Nolan. Peonin chloride, C<sub>28</sub>H<sub>33</sub>O<sub>18</sub>Cl, forms microscopic brown-red

needles with a bronze lustre. *Variation in the colour of flowers.* With H. Mallison. The variations in the colour of flowers depends upon (a) the presence of different anthocyanins in one plant and even in the same flower, (b) the different amounts of the colouring matter present, (c) the reaction of the cell sap, and (d) the presence of yellow pigments. The rose-coloured cornflower contains, in addition to cyanin, 0.72 per cent. of pelargonin in the fresh, 3.75 per cent. in the dry flower. A violet-red variety of pelargonium was found to contain principally cyanin. The deep brownish-red dahlia contains 19.4 per cent. of cyanin in the dried flower. The scarlet-red variety contains from 4 to 5 per cent. pelargonin. Examples are given of the variation of the anthocyanin content of different flowers. In percentage of the dry weight, cyanin makes up 0.7 of the blue cornflower, 13-14 of the Bordeaux-coloured variety, 2 of the rose, and about 20 per cent. in the dark red dahlia. Pelargonin makes 1 of the *P. peltatum*, 6-14 of the *P. zonale*, 4-5.6 per cent. of the scarlet-red dahlia. Peonin makes up 3-3.5 of the peony, malvin 6.4 per cent. of the *Malva-sylvestris*. Because of the amphoteric nature of anthocyanins, it follows that in red flowers they are present as acid salts, in the violet as neutral dyes and in the blue flowers as alkaline salts. These observations have been confirmed in a number of flowers. Among the yellow pigments may be met the indifferent carotinoids, principally carotin and xanthophyll, the flavone dyes coupled with a sugar or the dyes known to botanists as "anthochlor." Thus from the deep yellow variety of *Viola tricolor*, 25 per cent. of the dry weight was found to consist of violaquercitrin (rutin) while 0.6 per cent. was carotin. The following methods have been used for isolating the anthocyanins: (1) Precipitation and crystallization of the chloride: pelargonin and cyanin. (2) Purification of the dye and then crystallization of the chloride: (a) cyanin from the cornflower precipitated as the alkali salt from aqueous solution by EtOH. (b) Delphinin is precipitated as the violet colour base from dilute solution by pure EtOH. (c) Myrtillin is purified by repeatedly precipitating from an aqueous solution by HCl. (d) Delphinin is purified by heating with dilute HCl, hydrolyzing the impurities. (e) Cyanins from the rose and peonin are freed from impurities by long digestion with AcOH-MeOH-HCl. (3) Precipitation as the picrate and transformation into the chloride: enin, myrtillin, idaein. Cyanin was isolated from the dahlia by extracting 700 Gm. of the fresh



dark red flowers with AcOH, adding MeOH-HCl and precipitating with Et<sub>2</sub>O, giving 15 Gm. reddish-brown powder with a purity of 76. This is purified by dissolving in 700 c.c. of 7 per cent. HCl, filtering and allowing to cryst. Again recrystallized by dissolving in 750 c.c. hot water and adding an equal volume of 7 per cent. HCl, 7.4 Gm. pure chloride were obtained. Pelargonin was isolated from the dahlia by extracting 1.875 Kgm. fresh flowers with 3 parts AcOH for two days, treating the filtrate with 150 c.c. of 9 per cent. MeOH-HCl and precipitating with 10 litres of Et<sub>2</sub>O. The precipitate is crystallized from 500 c.c. 2 per cent. HCl after warming several hours at 60–70°.

The chlorides, picrates and hydrolysis products of all these anthocyanins are fully described.

#### **Bilirubin, Identification of Traces of, in Albuminous Fluids.**

A. A. H. vanden Bergh and J. J. Schlüter. (*Proc. K. Akad. Wetenschappen*, 1914, 17, 807; *Chem. Abstr.*, 195, 9, 1492.) To detect with certainty the presence of small quantities of bilirubin in blood serum or other albuminous fluid, 20 c.c. of pure acetone is added to 10 c.c. of the serum, and the more or less intensely yellow solution, after removal of the albumin precipitate, is evaporated *in vacuo* at the ordinary temperature until the acetone has been removed. The residual aqueous solution is treated with Et<sub>2</sub>O to remove the fatty substances, 2 c.c. of CHCl<sub>3</sub> is added, and the mixture is faintly acidified with HCl and well shaken. The CHCl<sub>3</sub> solution containing the bilirubin is thoroughly washed with water to remove all the HCl, and is dried, if necessary, with anhydrous Na<sub>2</sub>SO<sub>4</sub>. Bilirubin can be detected in the yellow CHCl<sub>3</sub> solution by the usual tests, by the reactions of Gmelin and of Ehrlich, and by the isolation of yellow, microscopic crystals.

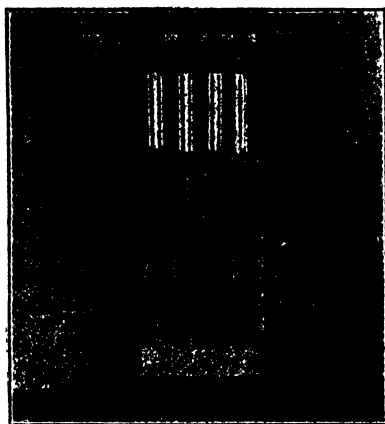
**Bixin, Formula of.** J. F. B. Van Hasselt. (*Rec. trav. chim.*, 33, 192; *Chem. Abstr. Amer. Chem. Soc.*, 1914, 8, 3016.) In a study of the constitution of bixin it was shown that the formula is C<sub>29</sub>H<sub>34</sub>O<sub>5</sub>, not C<sub>28</sub>H<sub>34</sub>O<sub>5</sub>. The bixin was first purified by means of AcMe which dissolves a quantity of resin containing impure orellin. After extracting with CHCl<sub>3</sub> in a Soxhlet the crystals are introduced into a tall cylinder and washed by decantation with AcMe, by which an amorphous substance is removed. The bixin was recrystallized and decanted three times and gave some of the amorphous compound each time,

which is probably an oxidation product of bixin formed during extraction. Heiduschka and Riffart's method does not give so good a product as shown by analysis. (See also *Y.B.*, 1910, 55; 1911, 54.)

**Carajura and Chica Red.** A. G. Perkin. (*Proc. Chem. Soc.*, 1914, 30, 212.) The material examined, supplied by Messrs. Wright, Layman and Umney, is a rare Central American pigment, prepared by the Indians from *Bigonia chica*. It is said to be the same as "Chica red" reported on by Erdmann in 1857. It consists of the Ca compounds of at least two colouring matters, which have been either precipitated on, or mixed with a substance resembling peat or ground bark. After treatment with HCl, EtOH removes the colouring matters as a resin; from this *carajurin* is removed by boiling  $C_6H_6$ . It separates in ruby crystals having the provisional formula  $C_{18}H_{16}O_5$ ; m.p.  $204^{\circ}$ – $206^{\circ}C.$ ; soluble in boiling alkali, giving a red solution nearly devoid of dyeing properties. It readily yields oxonium salts with mineral acids, which crystallize in bright orange needles. Of these, the sulphate, probably  $C_{18}H_{16}O_5H_2SO_4 \cdot H_2O$ , is the most stable. HI converts carajurin into *carajuretin* hydriodide, forming bright scarlet needles. By treating this with pyridine carajuretin is obtained also in scarlet needles, probably  $C_{18}H_{12}O_5$ , m.p. above  $330^{\circ}$ . Other compounds and decomposition products are described. The portion of the EtOH soluble resin, insoluble in  $C_6H_6$ , yields *carajurone* to  $Et_2O$ . This is a scarlet powder, readily assuming a beetle-green lustre and possessing strong dyeing properties. It contains more O than carajurin. A small amount of a similar but brighter lake, from British Guiana, obtained from the leaves of a "bush-rope" yield a colour dyeing with alizarin shades. It is considered also to be "Chica red" and seems to differ in some respects from "Carajura."

**Colorimetric Methods of Analysis.** F. E. Niece. (*Drugg. Circ.*, 1915, 59, 365.) Colorimetric methods for determining many chemical substances are advocated as being more rapid and sometimes more accurate than the ordinary gravimetric or volumetric processes of analysis. A simple and inexpensive colorimeter has been devised as shown in the illustration. The general method of colorimetry is outlined and stress laid on the necessity for extreme accuracy in weighing and measuring

in the preparation of standards. A number of substances which may be determined colorimetrically are enumerated.



A—Movable mirror. B—Reflection of colours in tubes. C—Upper framework movable. D—Nessler tubes. E—Screw hinge. F—Tube support with holes. G—Upright supports (rigid). H—Glass shelf tube rest. I—White opaque glass light reflector. J—Base for stand. K—Thumb screw for adjusting upper movable framework.

**Colouring Matters contained as Glucoside in the Flowers of some Indian Plants.** A. G. Perkin and I. Shulman. (*Proc. Chem. Soc.*, 1915, 30, 200.) These flowers, the description of which is given below, were specially collected in India, in the anticipation that they contained some quantity of dye-stuff. This has not proved to be the case, the amount of colouring matter isolated being extremely small, and not always sufficient for analysis. The method of examination in each instance has consisted of treating the concentrated EtOH extract with water, removal of suspended waxy matter, digestion of the aqueous solution with boiling HCl to hydrolyze the glucosides, and isolation of the colouring matter by means of Et<sub>2</sub>O. Incidentally, the use of NaHCO<sub>3</sub> solution has been found of much service in purifying minute amounts of these substances. To this the crude product dissolved in alcohol is added, and, after agitation, Et<sub>2</sub>O now removes a fairly pure compound.

*Poinciana regia* (Bengal).—Three hundred and fifty Gm. gave 0.31 Gm. of colouring matter, which crystallized in yellow needles, dissolved in alkalis with a yellow coloration, and proved

to be quercetin. The acetyl compound, colourless needles, m.p.  $194^{\circ}$ – $196^{\circ}$ , gave C = 58.75; H = 3.84 per cent. An extract of these flowers had practically no dyeing action on mordanted cotton. *Impatiens balsamina* (Chansili Pass) yielded but a trace of substance, evidently kaempferol. It separated from acetic acid in needles, m.p.  $275^{\circ}$ – $278^{\circ}$ , and gave with sulphuric acid a fluorescent solution. *Woodfordia floribunda*.—Seven hundred Gm. gave 0.84 Gm. of crude colouring matter, and this consisted of two substances readily separated by means of alcohol. The sparingly soluble compound (0.61 Gm.) crystallized from pyridine in needles, and was recognized as ellagic acid. The soluble colouring matter (yellow needles, m.p.  $285^{\circ}$ – $291^{\circ}$ ), of which but a minute amount was available, was possibly an impure quercetin. *Erythrina stricta* (vernacular name, "Konkathet") gave a trace of kaempferol.

The flowers of the common fuchsia, *F. macrostema globosa*, were found to yield traces of ellagic acid.

**Colours of Flowers as Indicators.** H. W. Brubaker. (*J. Amer. Chem. Soc.*, 1914, **36**, 1925–1928.) The colouring matter of the *Rosa rugosa* gives a green colour reaction with alkalies, red with acids, and is colourless at the intermediate point. It may be separated by triturating the petals with sand and 95 per cent. EtOH, filtering the extract, allowing the filtrate to evaporate spontaneously, and redissolving the residue with water. Addition of EtOH to bring the alcoholic strength to 50 per cent. is made to preserve the solution. The indicator behaves like phenolphthalein towards  $\text{CO}_2$ . In the form of test-paper it is sensitive to NaOH 1:25,000. The colouring matter of the *Lathyrus latifolius*, purple vetch and iris, behave in a similar manner, and appear to be closely related to the colouring matters of hollyhocks and dahlias. The petals of most flowers are changed in colour by alkalies, being reconverted to the original shade or changed to red by acids. The yellows are unaffected by either acid or alkali, while the white clover, white rose, white pansy, white geranium are changed to yellow by alkalies, and white again by acids. The reds and purples are changed to green or greenish-blue by alkali, while they are restored to their original colour or are made a brighter red by acids. Usually the natural colouring matters are acid, or in some cases neutral.

**Colours of Flowers, Investigation of.** P. Q. Keegan.

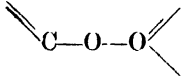
(*Chem. News*, 1915, 111, 87.) Plants producing phloroglucol tannins yield original reds only; plants producing caffetannin yield original blues only. Plants producing gallotannin seem inclined (owing to less acid) to display vivid violets or warm purplish blues according to circumstances, but never cold true blues. There is only one anthocyan pigment in plants producing caffetannin; the red on stem or petal is here due to acid only. Yellow flowers are due to carotin, or its diffused oxidized product (xanthophyll), and very rarely to a flavone. The white flower very often encloses a very dilute solution of anthocyan, but is due chiefly to the circumstance that intense deassimilation has not been occasioned in the corolla, because the nitrogen needed by the reproductive organs has been fully supplied from sources other than the inflorescence. Grafe's opinion that "a special chromogen of anthocyan cannot exist" is incorrect so far as the specific hereditary tendency of the protoplasm to form certain kinds of tannin is concerned.

**Dye Lichens, British.** A. Edge. (*J. Soc. Dyers' Colourists*, 30, 186; *Chem. Abstr.*, 1914, 8, 3369.) Archil and cudbear still find large employment in dyeing. They are produced by the combined action of  $\text{NH}_4\text{OH}$  and air upon *Rocella tinctoria* and *R. fuciformis*. *Lecanora tartarea* and *Urceolaria calcarea* were used chiefly by Scotch cudbear makers, and others capable of yielding purple to crimson dyes were of commercial value at one time although not equal in brilliancy to those obtained from the *Rocellae*. In some cases (*Parmelia* or *Crottes*) the dye seems to be already formed in the plant and is obtained by simple extraction. In this case the plant is gathered in July and August when richest in colouring matter and simply dried in the sun. They dye wool directly a brown shade although the addition of a small amount of  $\text{AcOH}$  to the bath produces better exhaustion. They do not form lakes with metals although their fastness to acids, alkalies, milling and scouring are increased by an after-treatment with  $\text{K}_2\text{Cr}_2\text{O}_7$  and  $\text{CuSO}_4$ . Fastness to light, however, does not seem to be thus improved.

**Fat Stains for Museum Specimens.** R. H. Malone and L. Hedrick. (*J. Path. Bact.*, 19, 102; *Chem. Abstr. Amer. Chem. Soc.*, 1914, 8, 356.) The tissues are cut in slices 1 to 2. cm. thick, and immersed in formaldehyde solution for 3 days, until fixed, hardened and partially bleached. They are then

washed in running water for 10 minutes, after which they are transferred to a saturated solution of Scarlet Red in 70 per cent. EtOH. The specimens should rest on cotton wool, and not on the bottom of the container, which should be tightly closed. The tissue should be trimmed ready for mounting before staining. The process must be stopped before the stain penetrates other than the fatty tissue. About 2 hours should suffice. After this the tissues are washed in running water for 10 minutes and then placed in a saturated solution of  $\text{HgCl}_2$  for half to one hour for differentiation. Mount in 5 per cent. formaldehyde solution.

**Flowers and Fruits, The Colouring Matter of.** R. Willstätter. (*Sitzb. preuss. Akad. Wissenschaften*, 1914, 402; *Chem. Abstr.*, 1914, 6, 3421.) The anthocyanins form a class of vegetable bases, in which the basic properties are due to oxonium O, in quinoid combination. They are distinguished, by the formation of stable oxonium salts, from the yellow flavone and flavonol groups, which are not present in the plants as oxonium compounds. They align themselves with compounds of the oxazine type. Their salts with organic and inorganic acids are red; on neutralization the colour becomes violet, with formation of an inner salt of the phenolbetaine type,

. The alkaline salts are blue, and are formed

without alteration of the inner oxonium salt group. They are glucosides. The anthocyan of the cornflower, *cyanin*, hydrolyzes into 2 mols. dextrose and 1 mol. of cyanidin. Careful analysis shows the formula of cyanidin to be  $\text{C}_{15}\text{H}_{11}\text{O}_6\text{Cl}$ , instead of  $\text{C}_{16}\text{H}_{13}\text{O}_7\text{Cl}$ , as formerly reported. The anthocyan of the rose is a diglucoside of this cyanidin, and that of the cranberry a galactoside, formed from 1 mol. of galactose and 1 mol. of cyanidin. The bleaching of anthocyan solutions is due to an isomerization analogous to the change of a  $\text{Ph}_3\text{CH}$  dye into its carbinol. Addition of acid reverses this reaction. Various anthocyanins were isolated by crystallizing their picrates. The anthocyan of purple grapes (*enin*) was separated by extraction of the skins with AcOH, precipitating as an oil with  $\text{Et}_2\text{O}$  and solution in picric acid. On cooling, long red prisms of picrate crystallize out. The hydrochloride crystallized from aqueous EtOH, in clumps of thick, beetle-green prisms. The anthocyan

(*myrtilin*) of the whortleberry was extracted from the dried skins with hot EtOH containing HCl, precipitated by Et<sub>2</sub>O, taken up with hot H<sub>2</sub>O and precipitated by addition of strong HCl; flat prisms from aqueous EtOH. The anthocyan of larkspur (*delphinin*) on hydrolysis gave 2 mols. dextrose, 2 mols. *p*-HOC<sub>6</sub>H<sub>4</sub>CO<sub>2</sub>H and 1 mol. *delphinidin*, C<sub>15</sub>H<sub>11</sub>O<sub>7</sub>Cl. The *pelargonin* of the scarlet pelargonium flower on hydrolysis gave 2 mols. dextrose and 1 mol. of *pelargonidin*, C<sub>15</sub>H<sub>11</sub>O<sub>6</sub>Cl. The *enin* of grapes gave 1 mol. of dextrose and 1 mol. of *enidin*, C<sub>17</sub>H<sub>13</sub>O<sub>7</sub>Cl. The *myrtilin* of the whortleberry contains another anthocyanidin, which is also present in the rose mallow\*. The absorption spectra are similar, with a broad band in the blue and green. The optical rotations are characteristic, and very high (200°–1400°). The anthocyanidins differ in colour, solubility and Fe<sub>2</sub>Cl<sub>6</sub> reaction. They are stable in the cold, but isomerize on heating. The reaction is reversed on adding acid. Structural formulæ, based on a pyrylium nucleus, are given for *cyanidin chloride*, *pelargonidin chloride*, *delphinidin chloride* and *enidin chloride*. The rearrangement to the colourless form takes place by reaction with H<sub>2</sub>O to form a carbinol or pseudo base. Direct reduction of flavones does not produce anthocyanins.

**Lycopersicin, the Red Pigment of the Tomato.** B. M. Duggar. (*Wash. Univ. Studies Chem. Abstr.*, 1915, 9, 1346.) The principal pigment of tomatoes is distinct from carotin, having a different absorption spectrum. In the mature fruit it is generally found in the form of needle-like crystals, but also sometimes in the form of long narrow bars and bacilloidal granules, and possibly irregular forms. The chemical rays of light were found to have little effect in making the fruit red. Temperatures higher than 30° inhibited the development of red pigment perhaps because of decreased acidity, but did not destroy it, since a return to normal temperatures resulted in rapid red pigmentation. At normal temperatures O appears to be necessary for reddening. Formation of lycopersicin follows destruction of chlorophyll, possibly involving increased permeability of cell structures. No correlation between lycopersicin development and oxidase content was found. (See also *Y.B.*, 1905, 144.)

**Myrica Rubens Bark, Colouring Principle of.** S. Satow. (*J. Ind. Eng. Chem.*, 1915, 7, 113.) The decoction of the bark of *Myrica rubens* has been used in Japan from ancient times

as a yellow or black dye. By extracting the coarsely powdered bark with a large volume of water and filtering while hot a crystalline glucoside was obtained on cooling. This was recrystallized from acetone, being obtained in greenish yellow monoclinic prisms,  $C_{15}H_{10}O_8 + H_2O$ . It contains 6 OH groups. It is identical or closely allied to the myricetin of A. G. Perkin. It dyes brownish yellow, orange yellow, orange brown, and black, with alumina, tin chrome or iron mordants.

**Rhamnus Catharticus, The Colouring Matters of.** J. O e s c h and A. G. P e r k i n. (*Proc. Chem. Soc.*, 30, 236.) According to Tschirch and Polacci (*Y.B.*, 1901, 106) the berries of *R. catharticus* contain (as glucoside) the yellow colouring matters rhamnocitrin,  $C_{13}H_{10}O_6$ , rhamnolutin,  $C_{15}H_{10}O_6$ , rhamnochrysin,  $C_{13}H_{10}O_7$ , and  $\beta$ -rhamnocitrin,  $C_{13}H_{10}O_6$ , in all of which methoxy-groups are absent. Krasowski (*Y.B.*, 1909, 75), on the other hand, could isolate only rhamnetin and quercetin, and considered that rhamnolutin is possibly rhamnetin, whereas rhamnocitrin is exactly like quercetin and rhamnetin. Again, instead of rhamnochrysin, a mixture of quercetin and emodin was obtained. The results of the present investigation show that rhamnocitrin,  $C_{16}H_{12}O_6$ , is a monomethyl ether of kaempferol; that rhamnolutin,  $C_{15}H_{10}O_6$ , the main colouring matter, is kaempferol, whereas  $\beta$ -rhamnocitrin present in small amount is rhamnetin,  $C_{16}H_{12}O_7$ . Incidentally, it was found that pure acetyl-rhamnetin melts at  $190^\circ$ – $192^\circ$ C. As indicated by Krasowski, quercetin can also be obtained from these berries, but the amount detected was trifling. The presence of a substance corresponding with rhamnochrysin could not be confirmed.

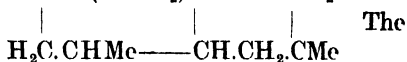
## ESSENTIAL OILS.

**Abies Concolor, Oil of Leaf and Twig, and of Bark.** A. W. S c h o r g e r. (*J. Ind. Eng. Chem.*, 1914, 6, 809.) The white pine, *Abies concolor*, occurs on mountain slopes from Southern Oregon to Lower California, and from Nevada, Utah, and Southern Mexico through Arizona and New Mexico. *Leaf and twig oil*.—There is considerable variation in the characters and constitution of various batches of this oil. The sp.g. ranges from 0.8720 to 0.8777;  $\alpha_D$  from  $-20.11^\circ$  to  $-27.94^\circ$ ;  $\eta_{D_{15}}$  from 1.4781 to 1.4796. The oils had the following average percentage composition: Laevo- $\alpha$ -pinene, 12; laevo-camphene, 8; laevo-



$\beta$ -pinene, 42; laevo-phellandrene, 15; esters (as bornyl acetate), 6.5; free borneol, 9.5; "green oil," 3. *Bark oil*.—This differed materially from the above. Sp.g. 0.8702 to 0.8767;  $a_D$   $-20.15^\circ$  to  $-20.95^\circ$ ;  $\eta_{D15}$  1.4809 to 1.4833. It contained the following percentages of the constituents named: Laevo- $\alpha$ -pinene, 9; laevo- $\beta$ -pinene, 60; dipentene, 12 to 13; ester (as bornyl acetate), 2.5; free borneol, 4.5; "green oil," 5. The "green oil" boils from  $265^\circ$ – $285^\circ\text{C}$ . It has the sp.g. 0.925 at  $19.5^\circ\text{C}$ .;  $a_{D20}$   $-0.49$ . Its presence imparts a yellow tint to all the original oils. Although the majority of these are fluorescent, the "green oil" is not so. Possibly its presence in the leaf and twig oil may be due to the bark occurring on the twigs.

**African Copaiba, Copaene from.** F. W. Semmler and H. Stenzel. (*Berichte*, 1914, **47**, 2555.) Schimmels obtained from African copaiba balsam oil a fraction (*copaene*),  $\text{C}_{15}\text{H}_{24}$ , b.p. under 10 mm.  $119$ – $20^\circ\text{C}$ ., sp.g. 0.9077,  $[\alpha]_D$   $-13.21^\circ$ ; this yields no solid nitrosochloride, but gives 30 per cent. of cadinene hydrochloride, while with H and Pt in  $\text{Et}_2\text{O}$  is obtained *dihydrocopaene*,  $\text{C}_{15}\text{H}_{26}$ , b.p. under 12 mm.  $118$ – $21^\circ\text{C}$ .,  $\eta_D$  1.47987, sp.g. 0.8926,  $[\alpha]_D$   $-12.2^\circ$ . The data indicate that copaene represents an entirely new type of tricyclic sesquiterpene to which is given the structure  $\text{HC} : \text{C}(\text{CHMe}_2) \text{CH}(\text{CH}-\text{CH}_2$ .



products of ozonization are chiefly acid, a viscous copaeneketo-acid, sp.g.  $215$ – $25^\circ$ , being isolated; semicarbazone, m.p.  $221^\circ\text{C}$ . Through the Ag salt was prepared the methyl ester,  $\text{C}_{13}\text{H}_{23}\text{COCO}_2\text{Me}$ , b.p. under 14 mm.  $177$ – $82^\circ\text{C}$ ., sp.g. at  $20^\circ\text{C}$ . 1.0243,  $\eta_D$  1.47694,  $[\alpha]_D$   $29.4^\circ$ ; semicarbazone, m.p.  $193.5$ – $4.0^\circ$ . With  $\text{KMnO}_4$  in aqueous AcMe (3 atoms O), the same acid was obtained, also a glycol,  $\text{C}_{15}\text{H}_{26}\text{O}_2$ , b.p. under 17 mm.  $178$ – $85^\circ\text{C}$ ., sp.g. at  $220^\circ\text{C}$ . 1.039,  $\eta_D$  1.50266,  $a_D$   $13^\circ$ . With Br in NaOH the acid gives copaenedicarboxylic acid,  $\text{C}_{13}\text{H}_{18}\text{O}_4$ , whose dimethyl ester b.p. under 17 mm.  $178$ – $82^\circ\text{C}$ ., sp.g.  $24^\circ\text{C}$ . 1.072,  $\eta_D$  1.47242,  $[\alpha]_D$   $34^\circ$ . (See also *Y.B.*, 1914, 50.)

**Artemisia Absinthium, Essential Oil of.** V. Paolini and R. Lo Monaco. (*Atti del Reale Accad. Lincei*, 1914, **23**, 123; *J.S.C.I.*, 1915, **34**, 247.) The oil distilled from fresh plants cultivated near Rome contained 10 per cent. of mixed

$\alpha$  and  $\beta$  thujone; 48 per cent. of total thujol, free and as acetic, isovalerianic and palmitic esters; a mixture of at least two compounds in which  $\delta$ -thujol predominated; with phellandrene, cadinene and a blue oil of unknown composition. (See also *Y.B.*, 1907, 3, 18, 178; 1910, 88.)

**Artemisia Arborescens**, Oil of. T. Jona. (*Annali Chim. appl.*, 1914, 2, 63; *J.S.C.I.*, 1914, 33, 1072.) About 200 kilos. of the dried tops of the plants collected shortly before flowering, yielded on distillation 1.14 kilos. of a deep blue oil: Sp.g. 0.9458 at 15°C., soluble in all proportions in EtOH 90 per cent. and 1:10 in EtOH 80 per cent., and remained clear at -15°C. The oil contained neither sulphur nor nitrogen compounds; saponification value, 29.3; ester value, 19.5; acetyl value, 50.0; distilled chiefly between 185° and 310°C. at normal pressure and between 90° and 210°C. under 33 mm. It contained a mixture of high-boiling hydrocarbons; formic, acetic, isovaleric, pelargonic, palmitic, and stearic acids, partly free and partly as esters; about 13 per cent. of  $\beta$ -thujone, and 13.94 per cent. of alcohols,  $C_{10}H_{18}O$  (thujol and probably a small quantity of borncol), of which 8.58 per cent. were in the free state and 5.36 per cent. as esters.

**Azulene, the Blue Constituent of Chamomile Oil.** A. E. Sherrn dal. (*J. Amer. Chem. Soc.*, 1915, 37, 167.) By taking advantage of its solubility in mineral acids, the author has succeeded in isolating the blue constituent of oil of chamomile and certain other essential oils. A very blue fraction was shaken out with one-fifth its weight of  $H_2SO_4$ , 63 per cent. The separated acid liquid, diluted with water, was shaken out with petroleum benzin until no more blue colour was removed. The deep blue benzin solution was then shaken out with  $H_3PO_4$ , 85 per cent., and the deep red acid solution drawn off. This was diluted with water and extracted with  $Et_2O$ . The residue, after evaporating the  $Et_2O$ , was the colouring substance which was purified by steam distillation and then by distillation *in vacuo*. The product which has been named *azulene* is a hydrocarbon  $C_{15}H_{18}$ ; it is an intensely blue viscous liquid, black when viewed in quantity; having a phenolic odour recalling that of thymol; sp.g. 0.9738 at 25°C.; distilling with some decomposition between 295°-300°C. under normal pressure and between 185°-195°C. under 25 mm. Soluble in most organic solvents, and in inorganic acids; in  $H_2SO_4$ , 60 to 65 per cent.,

it gives a yellow fluorescent solution; in  $\text{H}_3\text{PO}_4$ , 85 per cent., the solution is reddish yellow by transmitted light and intense apple green by reflected light. Concentrated formic acid dissolves it with a green colour: glacial acetic acid does not affect the blue tint. Azulene is strongly resistant to  $\text{H}_2\text{SO}_4$ . It may be heated on the water-bath with 94 per cent.  $\text{H}_2\text{SO}_4$  for 15 hours, and on dilution liberates blue oil. Azulene is readily oxidized, but strong alkali does not affect its colour. When treated with acetic anhydride and  $\text{H}_2\text{SO}_4$  a sulphonic acid, soluble in water, is formed. This affords a deep violet crystalline Na salt, which on heating gives off  $\text{SO}_2$  accompanied by violet fumes: the latter condense to a blue oil. The name azulene was originally proposed by Riese, in 1864, for the blue constituent of chamomile oil, which had not been then isolated.

**Azulene, Further Note on.** A. E. S h e r n d a l. (*J. Amer. Chem. Soc.*, 1915, 37, 1537.) The preparation of a picrate and its analysis; the preparation of artificial blue oils from various oils of the sesquiterpene type; the isolation and identification, by means of the picrate, of azulene from the blue fractions of the oils of cubebs, camphor, and from the artificial blue gurjun oil; the complete reduction of azulene to a colourless dihydro-sesquiterpene are described in detail. The picrate was prepared as follows: To a solution of 1.0 Gm. picric acid in 20 c.c. 95 per cent. EtOH, was added 1.0 Gm. azulene dissolved in 5 c.c. EtOH. In a few minutes the mixture set to a mass of crystals which dissolved again when warmed on the water-bath, and on cooling separated out in short, shiny, black needles. These were filtered off on the pump, and dried *in vacuo* over  $\text{H}_2\text{SO}_4$ . Yield, 0.73 Gm.; m.p.  $118^\circ\text{C}$ . The picrate dissolves readily in EtOH, acetone and  $\text{Et}_2\text{O}$  with a green colour; in  $\text{C}_6\text{H}_6$  and ethyl acetate with a blue colour. The green colour of the solutions is probably due to partial decomposition in the moist solvents. For the same reason, if dilute alcohol is used in the preparation, a substance with a lower m.p. is obtained, more or less contaminated by oil.  $\text{CCl}_4$  and benzin dissolve out the azulene and leave the picric acid undissolved. The picrate forms an excellent means of identifying azulene. To give an idea of the colour intensity of azulene, 0.064 Gm. of the pure hydrocarbon was dissolved in a litre of benzin, and an ammoniacal solution of copper sulphate made up to match it in

depth of colour. The latter solution contained 0.24 Gm.  $\text{CuSO}_4$  to the litre and matched the azulene solution almost exactly both in depth and shade of the colour, showing only a slightly less purple nuance. Azulene may be obtained by the action of strong  $\text{H}_2\text{SO}_4$  on certain sesquiterpene-containing oils in solution in glacial acetic acid such as the oils of gurjun balsam, guaiacum, amyris, and heavy eucalyptus sesquiterpene. The probable constitution of the molecule is discussed. It is certain that azulene is closely related to the sesquiterpenes and is tricyclic.

**Barosma Venusta, Oil of.** E. Goulding and O. D. Roberts. (*Proc. Chem. Soc.*, 1914.) The leaves of *Barosma venusta*, Eckl. and Zeyh., yield about 2 per cent. of an essential oil, which has a lemon-yellow colour, a pleasant odour, sp.g. 0.865, and  $\alpha_D^{22} + 0^\circ 47'$ . The percentage composition is approximately as follows: Hydrocarbons, chiefly or entirely myrcene, 43; aldehydes, chiefly or entirely anisaldehyde, 0.5; phenols, 0.2; methylchavicol, 21.4; alcohols, partly linalool (calculated as  $\text{C}_{10}\text{H}_{17}\cdot\text{OH}$ ), 14.3; esters (calculated as  $\text{C}_{10}\text{H}_{17}\cdot\text{OAc}$ ), 2.2; sesquiterpenes, etc., 18.4. (See also *Y.B.*, 1913, 216.)

**Black Sage, Oil of.** C. E. Burke and C. C. Scalione. (*J. Ind. Eng. Chem.*, 1914, 6, 804-6.) The constituents of the oil were determined as follows: Pinene 6 per cent.; cineol 30 per cent.; dipentene, terpinene, etc., 25 per cent.; thujone 8 per cent.; camphor 25 per cent.; resin 40 per cent.

**Bystropogon Mollis, "Argentine Mint," Oil of.** A. Doering. (*Bol. Acad. Ciencias Cordoba*, 19, 379; *J. Chem. Soc.*, 1914, 106 [1], 172.) The oil has the sp.g. 0.918-0.920 and contains as much as 2.5 per cent. of furfural. No menthol was detected; traces of phenolic bodies and 0.7 per cent. of free acids were found. The main fraction of the oil distils at 210. The terpene constituents have not been identified. The herb yields 0.4 per cent. of this oil.

**Calamus Oil, Japanese, Sesquiterpene from.** Y. Asahina and E. Imai. (*Jap. P.J.*, 1914, 393, 1257; *Chem. Abstr.*, 1915, 9, 1091.) A tricyclic terpene  $\text{C}_{15}\text{H}_{26}$ , a colourless oil with the odour of cedar; sp.g. 0.9379 at  $20^\circ$ ;  $\alpha_D - 2.06^\circ$   $\eta_{D20}$  1.51009, has been isolated from this oil.

**Calycanthus Floridus, Oil of.** E. R. Miller, G. W. Taylor and M. H. Eskew. (*J. Amer. Chem. Soc.*, 1914, **36**, 2182.) The stem and bark of *Calycanthus floridus* gave an essential oil containing *d*- and *l*- $\alpha$ -pinene, cineol, borncol, bornyl acetate and other esters, salicylic acid, and probably linalol. The cineol amount present varied from 36 and 69 per cent. in different specimens of the oil.

**Camphor, Determination of, in Tablets and Pills.** E. Doward. (*J. Ind. Eng. Chem.*, 1914, **6**, 489.) The camphor is steam distilled from the weighed material. A condenser with a specially wide tube is employed. The distillate is shaken out with  $C_6H_6$  and the amount of camphor determined by the optical deviation. Every  $0^{\circ}1'$  rotation = 0.01961 Gm. of camphor in 50 c.c. or 0.0098 Gm. in 25 c.c. of benzene. The factor for the conditions of working the method as above should be first determined with known quantities of camphor. (See also *Y.B.*, 1905, 55; 1910, 62; 1912, 77.)

**Camphor, Synthetic.** Ernst Richter. (*Apoth. Ztg.*, 1915, **30**, 14; *Chem. Abstr.*, 1915, **9**, 1366.) The chief impurities are bornyl chloride, camphene, borneol, isoborneol and EtOH. The first named may, if present, be detected to the amount of 0.5 per cent. as follows: Ignite a Cu gauze ( $1 \times 0.5$  cm.) until all evidence of halogen disappears, cool, then spread 0.05 Gm. of the sample thereon, ignite the camphor by bringing it for an instant into contact with the flame, but removing immediately therefrom and allowing it to burn until completely consumed. Now introduce the gauze into the Bunsen flame. A green colour indicates the presence of Cu halide. The test is so delicate that so small an amount as 0.00025 Gm. bornyl chloride may be detected.

**Camphor, Synthetic, and its Detection in Official Camphor.** Heffter; Langgaard; Bohrisch. (*Apoth. Ztg.*; *Pharm. Zentralhalle*; *Schweiz. Apoth. Ztg.* 1915, **53**, 101-2.) Unless synthetic camphor is free from impurities, e.g., pinene-HCl, camphene, borneol, and until experiments on animals have been extended to man, internal and subcutaneous uses of synthetic camphor should not be considered. Bohrisch brings forward the following test: To 0.1 Gm. of powdered camphor in test tube is added 2 c.c. of a 1 per cent. vanillin-HCl solution, and the mixture put into a beaker of water which is

warmed gradually. At 30°C. the colour is yellow, at 60°C. bluish green, at 75°–80°C. indigo-blue. The latter colour persists for several hours, even after cooling. Synthetic camphor by the same treatment gives only a yellow colour. Vanillin-HCl-H<sub>2</sub>SO<sub>4</sub> gives still better results. The best identity test is that of optical rotation, official camphor being dextro-rotatory; synthetic inactive. (See also *Y.B.*, 1904, 45; 1907, 30; 1908, 38.)

**Chamaecyparis Lawsoniana Wood, Essential Oil of.** A. W. Schorger. (*J. Ind. Eng. Chem.*, 1914, 6, 631.) The tree, known as Port Orford cedar, occurs on the Pacific Coast of Oregon and California. Picked pieces of resinous wood gave as much as 10 per cent. of oil; sp.g. 0.891;  $\eta_{D_{15}^{\circ}}$ , 477. When rectified it left a blood-red residuo. A specimen of oil 4 years old was rectified with Na<sub>2</sub>CO<sub>3</sub> solution. This rectified oil left no blood-red residue. It was also devoid of the marked physiological action on the kidneys shown by the unrectified oil; it had the sp.g. 0.8905 at 15°C.  $\eta_{D_{15}^{\circ}}$  1.4758;  $\alpha_{D_{15}^{\circ}}$  +39.60°; acid value 0.3; ester value, 32.8; acetyl value 71.57. It contains 60 to 61 per cent. of dextro  $\alpha$ -pinene; 6 to 7 per cent. of dipentene; 11 per cent. of free laevo-borneol and 11.5 per cent. of bornyl esters, calculated as acetate; also 6 to 7 per cent. of cadinene.

**"Chis-pine" Oil, Indian.** (*Perfumery Record*, 1915, 6, 94.) Puran Singh, in a report to the Indian Forest Department on this oil, published in the *Indian Forester*, suggests that its distillation should be carried out on the commercial scale since the oil may be used as a substitute for commercial "Siberian pine needle oil." Characters given by the author for this Indian product are as follows: Sp.g. at 20°, 0.874;  $\alpha_D$ , –6°15'; acid value, 1.03; ester value, 14.51; saponification value, 15.54; iodine value, 271.2; distils from 160°–165°, 23.8 per cent.; distils from 165°–170°, 29.0 per cent.; distils from 170°–180°, 19.8 per cent.; distils from 180°–200°, 15.1 per cent.; distils from 200°–215°, 6.7 per cent.; residue above 215°, 5.6 per cent. These five fractions had the following physical characters:—

Fraction.	Sp.g. at 20°.	$\alpha_D$ .
160°–165° . . .	0.860 . . .	–12°12'
165°–170° . . .	0.861 . . .	– 9°15'
170°–180° . . .	0.863 . . .	– 6°
180°–200° . . .	0.868 . . .	– 3°
200°–215° . . .	0.909 . . .	– 2°

The ester value of this oil is 14.5, which corresponds to about 5 per cent. of esters calculated as bornyl acetate, which is, of course, to be expected with an oil with the range of distillation temperatures indicated above; for bornyl acetate is sought for in essential oils in the fraction boiling at 220°–230°, which is practically non-existent in the oil in question. This Indian oil, therefore, does not in the least resemble the popular commercial Siberian pine oil, but appears to consist in the main of terpenes, and resembles in character the pine needle oils of the type of *Pinus sylvestris*, *Pinus pumilio* and various other oils of very low ester value which have been so largely superseded by Siberian pine oil.

**Cineol, Determination of, in Eucalyptus Oil.** J. L. Turner and R. C. Holmes. (*J. Amer. Pharm. Assoc.*, 1915, 4, 351.) Deliver from a pipette 10 c.c. of the oil into a glass dish (preferably a round bottom one) of 50 c.c. capacity, which is imbedded in finely cracked ice. Add 10 c.c. of concentrated arsenic acid (containing about 85 per cent.  $\text{As}_2\text{O}_5$ ; and stir until precipitation is complete. When the mixture ceases to congeal further, allow to stand for 10 minutes in the ice. At this point, if the mixture forms a hard mass, indicating an oil rich in cineol, 5 c.c. of purified petroleum ether should be added, and the mass mixed well; transfer immediately to a hardened filter paper by means of a pliable horn spatula; spread evenly over the surface of the paper and lay a second hardened filter paper over the top. Press these between blotting paper in a letter-press for 1 minute. Change the outside papers and press again, repeating the operation, if necessary, until the cineol arsenate is apparently dry, and separates readily when touched with a spatula. The pressing is *not* complete when a hard mass remains which is broken up with difficulty; the method usually requires two changes of filter paper, pressing each time for about two minutes; if left too long in the press the compound may decompose. Now transfer the compound completely by means of the horn spatula to a glass funnel inserted into a 100 c.c. Cassia flask with neck measuring 10 c.c. graduated in 1/10 c.c. Wash the precipitate into the flask with a stream of hot water, assisting the disintegration with a glass rod. Place the flask in boiling water and rotate until the compound is thoroughly broken up; add enough water to cause the cineol to rise into the neck of the flask, cool to room temperature and read off the volume of cineol.

$\text{As}_2\text{O}_3$  forms with cineol an addition compound which is sufficiently stable for all practical purposes. While the  $\text{As}_2\text{O}_3$  method cannot be regarded as quite scientifically exact, it gives results varying only slightly, and is superior to any method yet proposed for the determination of cineol. The results obtained are concordant within 2 per cent.

The resorcinol method should not be adopted for the U.S.P. IX. for it will unquestionably lead to the introduction into commerce of low grade eucalyptus oils; it would be far better to retain the present unsatisfactory  $\text{P}_2\text{O}_5$  method, which undoubtedly is responsible for the fact that the majority of eucalyptus oils at present on the market possess a high cineol content. Arsenic acid may be obtained in commerce in crystalline form; and may be dissolved in water in such proportion that the resulting solution has the sp.g. 2.173 at  $25^\circ$  (corresponding approximately to 85 per cent. arsenic acid), or it may be conveniently prepared as follows: Place in a porcelain evaporating dish 50 c.c.  $\text{HNO}_3$  sp.g. 1.142, and add 60 Gm.  $\text{As}_2\text{O}_3$  in small portions, stirring continuously; after the reaction becomes less violent, heat over Bunsen burner until the oxidation is complete and the excess of nitric acid is evaporated; test for freedom from both arsenic trioxide and nitric acid; filter and evaporate to about 100 Gm. The resulting solution contains about 85 per cent. of  $\text{H}_3\text{AsO}_4$ .

**Cineol, Determination of, by Means of the Resorcinol Method.**  
H. G. A. H a r d i n g. (*Analyst*, 1914, 39, 476.) The author confirms the unreliability of the resorcinol method when applied direct to natural oils. A case is cited in which a pure cineol-free oil from *Eucalyptus dives* indicated 32 per cent. of cineol with the resorcinol method. Nevertheless the method may be successfully applied to cineol containing fractions of eucalyptus oil, provided the amount of cineol present does not exceed 40 to 50 per cent. To dilute these fractions before applying the test, the fraction of ordinary turpentine distilling between  $156^\circ$ – $160^\circ\text{C}$ . is employed. One hundred c.c. of the oil to be tested for cineol is distilled in a 150 c.c. flask, reserving the distillate collected from  $170^\circ$ – $190^\circ\text{C}$ ., and then diluting to 100 c.c. with the distilled turpentine. If a trial shows the percentage of cineol to be above 70 per cent., the cineol fraction is diluted further with the turpentine, so that the percentage is not over 50. The temperature is noted, and 6 to 10 c.c. shaken with warm 55 per cent. resorcinol solution [in a Hirschsohn flask]. After five minutes'



shaking more solution is added, so as to bring the oil to the graduated neck. It is then cooled, and the volume read. (See also *Gen. Index* and *Y.B.*, 1908, 52, 53, 77; 1911, 60, 62; 1913, 72, 73.)

**Citronella Oil, Javan, Abnormal.** (*Perfum. Record*, 1914, 5, 275.) Specimens of Javan citronella oil having abnormal characters have been met with. These are marked by a distinct fishy odour, or a preponderance of that of geraniol. These oils had the following characters: sp.g., .899 to .901;  $\alpha_D$ ,  $-1^\circ$  to  $-2^\circ$ ; total acetylatable constituents, 79.5 to 82.5 per cent.; geraniol (Dupont and Labaune's method), 50 to 58 per cent.; citronellal by difference, 22 to 32 per cent.

The characters of normal Javan oils are: sp.g., 0.884 to 0.897;  $\alpha_D$ , about  $+1^\circ$ ; total acetylatable constituents, 92 to 94 per cent.; geraniol (Dupont and Labaune's method), 50 per cent.; citronellal by difference, 44 per cent. The cause of this departure from type is not known. They may be due to the condition or botanical source of the grass used for distillation, or to some defects of the latter process. The above results emphasize the importance of the total acetylatable constituents in the valuation of this oil.

**Clove Oil and Clove Leaf Oil, from Mauritius.** (*Bull. Imp. Inst.*, 1914, 12, 232.) *Clove oil.*—Two samples had the following characters: sp.g., 1.061 and 1.067;  $\alpha_D$ ,  $-0^\circ 10'$  and  $-0^\circ 5'$  at  $22^\circ\text{C}$ .; eugenol, 89 and 95 per cent.; solubility in EtOH 70 per cent., 1:1.5 and 1:1.25 or more. The yield of oil in Mauritius was small, 10.2 to 11.5 per cent. Possibly the cloves were not so dry as those used in Europe for distillation, which yield from 15 to 18 per cent. *Clove leaf oil.*—Three samples of oil had the sp.g. ranging from 1.059 to 1.065;  $\alpha_D$ ,  $-0^\circ 28'$  (one sample only); eugenol, from 90 to 91 per cent.; solubility in EtOH 70 per cent., 1:1.25 and more. The yield obtained was small, 1.62 per cent. Clove leaves from Seychelles distilled in England have given 4.5 per cent. of oil.

**Clove Fruits, "Mother of Cloves," Essential Oil of.** (*Bull. Imp. Inst.*, 1914, 12, 338.) A sample of "Mother of cloves" from Zanzibar, distilled at the Imperial Institute, gave 6.5 per cent. of oil; sp.g., 1.056;  $\alpha_D$ ,  $-0^\circ 33'$ ; eugenol, 90 per cent. The sample contained a considerable number of clove flower buds (normal cloves) as well as fruits.

**Cochlearia Officinalis, Oil of.** Van Piepenbrock and Pinkhof (*Pharm. Weekbl. ; Drugg. Circ.*, 1914, **58**, 667) have examined the oil of scurvy grass and have found the following constants : sp.g., 0.959 ;  $\eta_{D_{15}}$  1.4935 ;  $\alpha_D$  in 3 per cent. EtOH solution 70°4'. The oil was soluble in 1.9 parts by weight of EtOH 90 per cent. and contained 94 per cent. of butyl-iso-sulphocyanide. The latter is determined by a method similar to that applied for estimating allyl-iso-sulphocyanide in mustard oil. For making *Spiritus cochleariae* the authors recommend using the flowering tops of the plants which are richest in butyl-iso-sulphocyanide, 100 Gm. yielding a distillate which contains 41 Mgm. of the cyanide. The distillate from the same quantity of stems, leaves and herbs yield only 1.3, 3.1, and 4.5 Mgm. respectively.

**Costus-Root Oil, Constituents of.** F. W. Semmler and J. Feldstein. (*Berichte*, 1914, **47**, 2687.) Costus-root oil from *Saussurea lappa* has the following approximate constitution : Camphene, 0.4 ; phollandrene, 0.4 ; an unidentified terpene alcohol,  $C_{10}H_{16}O$ , 0.2 ; two sesquiterpenes,  $\alpha$ -costene 6.0 and  $\beta$ -costene 6.0 ; apilotaxene, 20 ; costol, 7 ; dihydrocostusolactone 15 ; costusolactone, 11.0 ; and costusic acid, 14.

**Cuscut Oil.** Puran Singh. (*Chem. and Drugg.*, 1914, **85**, 255.) After a summary of published literature of the subject, the results are given of the distillation of 7 samples of roots from different localities in India. The percentage of oil from these ranged from 0.37 to 1.14 per cent. These results were obtained from the steam distillation of 25 Gm. of material, and shaking out the aqueous distillate with  $CHCl_3$ . Good roots should yield from 0.7 to 1.0 per cent. of oil. The roots for distillation are collected in India towards the end of January. The yield of oil is always much lower from roots collected during the rainy season. A specimen of the oil rectified by steam distillation had the following characters : sp.g. at 15°C., 1.011 ;  $\alpha_D$ , -30.65 ;  $\eta_D$  20°C., 1.5165 ; acid-value, 10.5 ; ester-value, 69.6 ; acetyl, 132.8 ; hübl value, 194.4 ; soluble 1 : 2 in EtOH 80 per cent.

The residue, a dark red brittle resin, had a very high dextro-rotation (+488.04° ?). The rectified oil, it will be noted, is laevorotatory. Cuscut oil is generally recorded as having the  $\alpha_D$  +25 to +40. The laevorotation of the rectified oil is doubtless due to the removal of the highly dextrorotatory resin.

**Daniella Thurifera Oil, Cadinene in.** W. Lenz. (*Berichte*, 1914, 47, 1989.) The thin dark brown balsam yielded by the leguminous Togo-land plant, *Daniella thurifera*, contains cadinene. This is the first record of its occurrence in one of the *Leguminosae*. It has been found in *Anonaceae*, *Burseraceae*, *Coniferae Labiatae*, *Lauraceae*, *Meliaceae*, *Piperaceae*, *Rutaceae* and *Umbelliferae*.

**Elsholtzia Cristata, Essential Oil of.** Y. Asahina and Y. Murayama. (*Archiv. Pharm.*, 1914, 252, 435.) Steam distilled, the dry labiate yields 2 per cent. of a yellowish, mobile oil with a peculiar odour. Sp.g. 0.970,  $\alpha_D -2.7$ , acid number 0, saponification number acetyl 14.84, b.p. 210–15°; the oil contains no aldehyde, phenol or MeO, but consists mainly of *elsholtzia ketone*,  $C_4H_2O(Me)(CO.CH_2CHMe_2)$ , nearly colourless, very mobile optically inactive liquid of peculiar aromatic odour, sp.g. 0.9817, b.p. 210°,  $\eta_D^{21}$  1.48424. It contains no OH, but yields with  $KMnO_4$  isovaleric acid, with Zn-Hg and HCl a colourless liquid,  $C_{10}H_{16}O$ , in small quantity, which has the odour of its ketonic source and does not react with semicarbazide. The ketone is converted in abs.  $Et_2O$  by Na and  $AmNO_2$  into *elsholtzia acid*,  $C_6H_8O_3$ , crystallizing in needles, m.p. 134°, sublimes on heating, yields on warming with a trace of isatin a reddish violet colour.

**Essential Oils, Hydrogen Number of Certain.** A. R. Albright. (*J. Amer. Chem. Soc.*, 1914, 36, 2188.) The author proposes to measure the hydrogen absorption of the unsaturated constituents in essential oils, using colloidal Pt. as the catalyzer. The method is fully described and the elaborate apparatus at present needed is figured. Results obtained with the oils of sassafras, anise, fennel, clove and pimente are quoted.

**Essential Oils, U.S.P., Seasonal Variation in the Characters of.** F. R. Bak. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1670.) Results are enumerated with the essential oils named, distilled at the Arlington Experimental Farm during the years 1907, 1908, 1909, 1910. *Colour* is found to vary very markedly in different years. It has little value as a critical character. *Odour* and *taste* vary also, but to a less degree.

The sp.g. of the oils investigated is given in the following table:—

The  $\alpha_D$  during the same period was thus recorded :—

Oils.	Requirements of U.S.P., 1910, 9th Revision 25°C.	Oil distilled during several successive seasons at Arlington Experimental Farm.			
		1907	1908.	1909.	1910.
<b>Chenopodium.</b>	0-955 to 0-980	0-933 (25°C.)	0-944 (24°C.)	—	—
<b>Fennel.</b>	0-953 to 0-973	—	—	0-927 (23°C.)*	0-944 (24°C.)
<b>Lavender.</b>	0-875 to 0-888	—	0-890 (24°C.)	0-892 (20°C.)	0-8962 (23°C.)
<b>Pennyroyal.</b>	0-920 to 0-935	—	0-9195 (24°C.)	0-9209 (22°C.)	0-9365 (23°C.)
<b>Peppermint.</b>	0-894 to 0-914	0-9048 (25°C.)	0-924 (23°C.)	0-9203 (22°C.)	0-9273 (23°C.)
<b>Rosemary.</b>	0-894 to 0-912	—	—	0-8947 (21°C.)	—
<b>Spearmint.</b>	0-914 to 0-934	0-923 (25°C.)	0-918 (22°C.)	0-9289 (21°C.)	0-9252 (24°C.)
<b>Thyme.</b>	0-894 to 0-929	0-898 (25°C.)	0-897 (24°C.)	0-9144 (21°C.)	0-936 (24°C.)

\* Oils from fruiting tops.

Oils.	Requirements of U.S.P., 1910, 9th Re- vision 100 mm. tubes.	Oil distilled during several successive seasons at Arlington Experimental Farm.			
		1907	1908.	1909.	1910.
<b>Chenopodium.</b>	—4° to —10°	—7.7° Specific rotation	—6.6° Specific rotation	—	—
<b>Fennel.</b>	+12° to 24°	—	—	+14.9° (50 mm. tube)*	+13.8° (50 mm. tube)*
<b>Lavender.</b>	—1° to —10°	—	—4° (50 mm. tube)	—6.5° (50 mm. tube)	—1.4° (50 mm. tube)
<b>Pennyroyal.</b>	+17° to 28°	—	+34.4° (50 mm. tube)	+28.6° (50 mm. tube)	+13.3° (50 mm. tube)
<b>Peppermint.</b>	—20° to 35°	—10.4° (50 mm. tube)	—4.5° (50 mm. tube)	—10.6° (50 mm. tube)	—5.7° (50 mm. tube)
<b>Rosemary.</b>	—	—	—	—2.4° (50 mm. tube)	—
<b>Spearmint.</b>	—35° to 50°	—45.6° (Specific rota- tion)	—27.3° (50 mm. tube)	—16.1° (50 mm. tube)	—20.3° (50 mm. tube)
<b>Thyme.</b>	Slightly laevogy- rate	—1.3° (50 mm. tube)	—0.6° (50 mm. tube)	—1.1° (50 mm. tube)	—

\* Oil from fruiting tops.

The solubility in EtOH 80 and 90 per cent. was found to be as follows:—

Oils.	Requirements of U.S.P., 1910, 9th Revision.	Oils distilled during several successive seasons at Arlington Experimental Farm.			
		1907.	1908.	1909.	1910.
Chenopodium	8 vols. 70% alcohol	1 vol. 80% alcohol clear solution	0.75 vol. 80% alcohol clear solution	—	—
Fennel . .	8 vols. 80% alcohol, 1 vol. 90% alcohol	—	—	*Turbid in 12 vols. 80% alcohol. 2 vols. 90% alcohol with clear solution	*2.5 vols. 80% alcohol. Clear in excess
Lavender .	3 vols. 70% alcohol	—	0.75 vol. 80% alcohol clear solution	1.2 vols. 80% alcohol	0.8 vol. 80% alcohol; turbid on 1 vols. or more
Pennyroyal.	2 vols. 70% alcohol	—	1 vol. 80% alcohol slightly turbid on further dilution	0.5 vol. 80% alcohol clear solution	All proportions in 80% alcohol
Peppermint	4 vols. 70% alcohol with only slight opalescence	1.5 vols. 80% clear solution	1.25 vols. 80% alcohol clear solution	1.2 vols. 80% alcohol; turbid with 2 vols. or more	0.6 vol. 80% alcohol; turbid with 2 vols. or more
Rosemary .	10 vols. 80% alcohol	—	—	Insoluble in 80% alcohol with clear solution. 0.75 vol. 90% alcohol	—
Spearmin <sup>t</sup> .	1 vol. 80% alcohol; cloudy on further dilution	1 vol. 80% alcohol clear solution	Turbid in all proportions with 80% alcohol. 1 vol. 90% alcohol cloudy on further dilution	1 vol. 80% alcohol; turbid with 2 vols. or more	0.4 vol. 80% alcohol; turbid with 2 vols. or more
Thyme . .	2 vols. 80% alcohol	1 vol. 80% alcohol clear solution	2 vols. 80% alcohol with turbidity. 1 vol. 90% alcohol, faint turbidity when further diluted	1.6 vols. 80% alcohol; clear on further dilution	1 vol. 80% clear on further dilution

\* Oils from fruiting tops.

It is unfortunate that the solubilities of all the oils in question were determined with 80 per cent. and 90 per cent. alcohol, the Pharmacopœial requirements specifying 70 per cent. alcohol in several cases. However, the results will at least admit of comparisons being made of the solubility of the oils from one season to another.

These results emphasize in a marked manner the profound influence of climatic and seasonal conditions on the physical characters of essential oils.

**Essential Oils of the Grass Family.** E. K r e m e r s. (*Drugg. Circ.*, 1915, 59, 355.) A résumé of the involved literature of the subject of the oils of the different species and varieties of the genera *Cymbopogon* and *Andropogon*. The following are the botanical sources of the oils named. *Cymbopogon martini* var. *motia*, Palma rosa oil; *C. martini* var. *sofia*, Ginger-grass oil; *C. flexuosus*, East Indian lemon-grass oil; *C. citratus*, West Indian lemon-grass oil; *C. nardus*, Ceylon citronella oil; *C. nardus* var. *linnaeri* (*typicus*), Java citronella oil; *C. nardus* var. *confertiflorus*, Java lemon-grass oil; *C. schoenanthus*, Camel-grass oil; *C. polyneuros*, Delft-grass oil; *C. coesius*, Kamakshi oil. The root oil of vetiver, and Samoan mumuta oil from the undetermined species of *Andropogon* are alluded to. The chemistry of all these, as shown in current literature, is dealt with.

**Esters, Determination of, in Essential Oils.** J. N i v i è r e. (*Bull. Soc. Chim.*, 1914, 15, 677.) The statement of Béhal that esters should be saponified in closed vessels to obtain accurate results is not substantiated. Concordant results were obtained by saponification in an open flask fitted either with an air tube or a reflux condenser. Higher results were obtained with closed flasks than with open flasks. Lavender oil gave a saponification value of 114.2 in an open flask and 117 in a closed flask; bergamot oil, open flask 104.1, closed flask 150.7. Pure linalyl acetate gave practically identical results either in open or closed flasks, viz. 242, but linalyl acetate containing 2 per cent. of œnanthol gave saponification values, open flask 239.8–239.9, closed flask 241.7–242. The differences therefore appear to be due, in part to the presence of aldehydes and not to the loss of volatile esters in the open flasks.

**Eucalyptus Globulus Oil, Californian.** C. E. B u r k e and C. C. S c a l i o n e. (*J. Ind. Eng. Chem.*, 1915, 7, 206.) The oil

distilled from *Eucalyptus globulus* in California has not the same characters as that distilled in Australia and does not meet the requirements for eucalyptus oil of the U.S.P. It has the sp.g. 0.9052 at 20°C.,  $\alpha_D + 14.42'$ ,  $\eta_D 1.46055$ ; and contains aldehydes, 6 per cent.; pinene, 21 to 22 per cent.; cineol, 47 per cent.; alcohols, including globulol and eudesmol, 23 per cent. The actual constituents of the oil are not different from those found in Australian oil, but the proportion in which they occur is very different, notably the large amount of pinene. This is the cause of the divergence of the characters.

**Eupatorium Capillifolium, E. Serotinum and some other Species of Eupatorium, Essential Oils of.** E. R. Miller. (*Bull. Univers. Wisconsin* [693]; *Drugg. Circ.*, 1915, 59, 225.) *Eupatorium capillifolium* gave from 0.36 to 1.11 per cent. of essential oil. Sp.g., 0.919 to 0.960; acid value, 0.10 to 0.25; saponification value, 5.6 to 16.68. The oil contained from 46 to 67 per cent. of thymohydroquinone dimethyl ether, phellandrene, a sabinene, and a small amount of linalol. *Eupatorium serotinum* gave 0.57 per cent. of oil. Sp.g., 0.9075;  $\eta_D 1.449$ ;  $\alpha_D - 7.36^\circ$ ; acid value, 0.508; saponification value, 28.7. It gave indications of the presence of phenolic constituents, but consists mainly of one or more sesquiterpenes. Neither *E. purpureum* nor *E. hyssopifolium* yielded any appreciable quantity of essential oil. From *E. perfoliatum*, only 10 minims was obtained from 30 pounds of material.

**Galbanum Oil, Characters and Constituents of.** F. W. Semmler and K. G. Jonas. (*Berichte*, 1914, 47, 2068.) The oil examined had the following characters: b.p. 55°–195°C. under 15 mm.,  $\eta_D = 1.49395$  at 25°C.,  $\alpha_D = +8^\circ$  at 25°C., sp.g. 0.9353. It contained pinene, cadinene, nopinene, myrcene, a terpene derivative,  $C_{10}H_{16}O$ , and a new sesquiterpene alcohol cadinol  $C_{15}H_{26}O$ . The terpene derivative boiled at 105°–115°C. at 15 mm., had the sp.g. 0.951 at 20°C.,  $\eta_D = 1.4918$  and  $\alpha_D = +6^\circ$ . Cadinol, boiled at 155°–165°C. at 15 mm., had the sp.g. 0.9720 at 20°C.,  $\eta_D = 1.50702$ , and  $\alpha_D = +22^\circ$ .

**Helichrysum saxatile (Moris.), Oil of.** L. Francesconi and E. Sernagiotto. (*Gaz. Chim. Ital.*, 1914, 44, II, 419; *J.S.C.I.*, 1915, 33, 1222.) The yellowish oil, obtained from plants growing in Sardinia, has a pleasant pungent odour, which when greatly diluted resembles that of the rose. It has the

sp.g. 0.9020,  $\eta_D=1.4769$ ,  $\alpha_D-11.71$  (in alcoholic solution), and distils chiefly at  $240^\circ\text{C}$ .

**Hops, The Aroma of.** J. Schmidt. (*Compt. rend. Lab. Carlsberg*, 1915, 11, 149; *Chem. Abstr.*, 1915, 9, 1658.) In all districts where the male hops have not been entirely eradicated, large stocks of hops will, in the long run, become deteriorated. Among the hop plants cultivated in the experimental garden of the Carlsberg Laboratory, there were two American plants, "Oregon Cluster" and "New York Spaulding English Cluster," the hops of which exhibited a very peculiar, turpentine-like aroma, so widely different from that of all European varieties, that a single hop could at once be recognized by the smell among hundreds of others. Cultivation experiments made during 1911-4 showed that this aroma remained apparently constant in the foreign climate in these two plants and their cones. Crossing experiments with the two American plants and Danish males showed that the turpentine-like aroma was transmitted to between 0.5 to 0.75 of the offspring plants. From these experiments and several others it would seem that the aroma of hops is not so "volatile" a character or so entirely due to purely local conditions as has generally been believed.

**Hops, Oil of, with Relation to the Geographical Sources of the Hops.** F. Rabak. (*J. Agr. Research*, 2, 115; *Chem. Abstr.*, 1914, 8, 3837.) The volatile oil of hops has been shown to consist chiefly of the terpene myrcene; heptolic, octolic, and nonoic acid esters of myrcenol; and the sesquiterpene, humulene, with traces of free acids,  $\text{CH}_2\text{O}$ , and probably some free alcohols. The several oils examined were found to contain varying proportions of the esters as well as of myrcene and humulene. Important differences are apparent not only during any particular season but for several seasons. These differences are shown in curves giving the physical and chemical properties of the oil. From the results obtained it is thought that the geographical source of hops may be indicated by the ester in the oil, since the ester values of the oils from hops of any particular source or season are very similar.

**Isomeric Linalols and the Scission of Inactive Linalol into its Optical Antipodes.** V. Paolini and Laura Divizia. (*Atti accad. Lincei*, 1914, 23, II, 171; *Chem. Abstr.*, 1915, 9, 1323.) Linalol,  $\text{C}_{10}\text{H}_{18}\text{O}$ , is a tertiary alcohol widely occurring



in nature; *d*-linalol predominates in coriander oil and *l*-linalol predominates in the oils of linalol, bergamot, neroli, etc. *d*-Linalol is isolated by fractionation of the saponified oil; it is purified by esterifying with phthalic anhydride and saponifying the acid phthalate. The product thus obtained has the b.p. 197°–200°, sp.g. 0.8622 to 0.875,  $\alpha_D$  –1.40° to –20°70' and for the dextro-isomer  $\alpha_D$  19°18'. All these mixtures are racemates of the two forms with either *d*- or *l*- in excess. Inactive linalol is not found in nature but has been synthesized in at least four ways. Since it does not form solid derivatives its complete purification is not easy. The authors find that the combination of the acid phthalic esters of linalol (and of santalol) with strychnine form crystalline compounds of different solubilities. By fractional crystallization of these strychnine compounds and subsequent regeneration of the alcohol, laevo and dextro-linalol (and santalol) are obtained in a pure state. Myrcenol of Power and Kleber is shown to be distinct from linalol.

**Italian Wormwood Oil, Constituents of.** V. Paolini and R. Lomonaco. (*Atti accad. Lincei*, 1914, 23 [2], 123; *Chem. Abstr.*, 1915, 9, 1323.) The fresh Italian herb of *Artemisia absinthium* gave 0.46 per cent. of greenish essential oil. About 10 per cent. of the oil was a mixture of  $\beta$ -thujone with some  $\alpha$ -thujone. This mixture is the "absinthol" of previous workers. About 48 per cent. of the oil consisted of thujyl alcohol, free or as esters.

**Java Citronella Oil, New Oxide in.** K. E. Spornitz. (*Ber.*, 1914, 47, 2478.) A fraction of citronella oil residues, b.p. 190°–200°C., when rectified over Na, gave *dicitronella oxide*  $C_{20}H_{34}O$ , b.p. 182°–183°C.,  $d_{20}^{20}$  0.9199,  $\eta_D$  1.49179,  $\alpha_D$  –4°, unchanged by Na and EtOH, reduced by H and Pt sponge in AcOH or Et<sub>2</sub>O to the tetrahydro compound, b.p. under 11.5 mm. 180°–185°C.  $d_{20}^{20}$  0.9001,  $\eta_D$  1.47457,  $\alpha_D \pm 0^\circ$ , while with HCl in Et<sub>2</sub>O is obtained the hydrochloride,  $C_{20}H_{35}OCl$ , tables from Et<sub>2</sub>O, m.p. 107.5°C., decomposed by boiling alcohols, converted by boiling alcoholic KOH into iso-di-citronella oxide, b.p. 176°–180°, sp.g. 0.9518,  $\eta_{20}$  1.49692,  $\alpha_D$  1°; the dihydro-compound, obtained from the above hydrochloride with Na and EtOH, m.p. 71°C., does not decolourize Br. The synthetic oxide prepared from citronellal by Semmler and Jonas agrees with *dicitronella oxide* in its physical constants, even the  $\alpha_D$  being

the same, but opposite in sign, but the author has been unable to prepare a hydrochloride of it corresponding to that of di-citronella oxide, and that the two compounds are identical seems yet improbable.

**Juniperus Oxycedrus Wood, Essential Oil of, for Therapeutic Use.** R. Huerre. (*Bull. Soc. Thérapeut.*; *L'Union Pharm.*, 1915, 56, 12.) The wood of *Juniperus oxycedrus* yields to steam distillation from 3 per cent. of essential oil in the autumn to 1.6 per cent. in the spring. This oil is a viscous dark yellow liquid; sp.g. 0.925;  $\alpha_D - 31.42'$  boiling between  $330^\circ - 360^\circ\text{C}$ . at ordinary pressure. Since it has a pleasant odour, between that of cedar and juniper oils, it is suggested for medicinal use in place of empyreumatic cade oil, since the powerful odour of the latter is considered to be very objectionable by many patients. The essential oil is said to be very efficacious in such cases. If it is desired to employ phenolic constituents similar to the 5 to 7 per cent. of total phenols which occur in cade oil, guaiacol and ethyl-, methyl- and propyl-guaiacols to that total may be dissolved in the essential oil. The sulphur compound of the oil is also suggested for use in dermatological practice. It is obtained by heating a mixture of the essential oil with precipitated sulphur at below  $300^\circ\text{C}$ . Chemical action ensues, with generation of heat and evolution of  $\text{H}_2\text{S}$ . The sulphurated product is a greenish oil containing from 2 to 3 per cent. of S, and having a slight odour of  $\text{SO}_2$ .

**Kaempferia Ethelae Tubers, Essential Oil of.** E. Goulding and Roberts. (*Bull. Imp. Inst.*, 1915, 13, 15.) The plant, known as "Sherungulu," is plentiful in the N.E. Transvaal. The tubers yielded 2 per cent. of essential oil calculated on the dry material. This was yellowish liquid with a pleasant odour, somewhat like neroli with a less pleasant odour recalling that of crushed ivy leaves. The oil had the sp.g. 0.943;  $\alpha_D + 19.47'$ ; ester value, 5; acetyl value, 47.6. The portion of the oil boiling above  $270^\circ\text{C}$ . deposited crystals which when recrystallized from EtOH formed large transparent diamond-shaped crystals, m.p.  $102^\circ\text{C}$ ., becoming brown on exposure. These have the odour of ivy leaves noticed in the oil. This is an unsaturated ketonic compound and is present in the proportion of 13 per cent. The following other constituents were isolated in the percentages given: terpenes (dipentene and probably pinene), 21.8; cineol, 17.2; alcohols (including linalol), 11.2; esters (chiefly

methyl-anthranilate), 1.3; phenols, 0.5; acids (chiefly or entirely acetic acid), 0.1; residue (probably chiefly sesquiterpenes), 34.9.

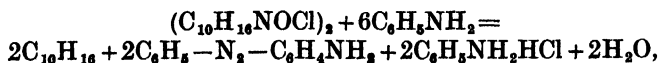
**Lantana Camara, Oil from the Flowers and Leaves of.** D. D. K a n g a. (*Arch. Pharm.*, 1914, 252, 1; *J.S.C.I.*, 1914.). *Lantana camara*, which is very abundant in Southern India and is known as "Ghaneri," originally came from Central America. The oil obtained from either the dry or fresh flowers or the leaves has a strong persistent but pleasant odour of sage, the yield being 0.02 per cent. from the dry flowers and 0.2 per cent. from the leaves. The characters of the oil from the dried flowers were: sp.g. 0.915 at 26°/15°C.,  $\eta_D$  1.4987 at 26°C.,  $a_D$  +23.9° at 26°C., saponification value 10, acetyl value 43.6. The oil from the leaves had the sp.g. 0.92114 at 24°/24°C.,  $\eta_D^{27}$  +1.48933,  $a_D$  +1.96°; this was obtained by the steam distillation of an alcoholic extract.

**Lavandula dentata, Essential Oil of.** Giessler. (*Perfum. Record*, 1915, 6, 214.) The botanical source of Spanish lavender oil, formerly attributed to *Lavandula stoechas*, is *L. dentata*. This oil has the following characters: sp.g. at 15° = 0.9620;  $a_D$  = +35°30';  $\eta_{D20}$  = 1.47909; acid value = 5.16; ester value = 13.1; acetyl value = 67.9. Dextro-camphor and dextro-fenchone are constituents of the oil, as well as, probably, fenchyl alcohol.

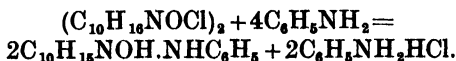
**Lavender Oil, Determination of Added Acetin.** F. D. D o d g e. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1664.) 3 Gm. oil is shaken with 75 c.c. water, in a 100 c.c. flask. The free acid is then titrated with aqueous N/2 KOH, and phenolphthalein. 0.5 c.c. more alkali is added, and the flask is heated at about 80°C., with frequent shaking. If the colour disappears, another 0.5 c.c. is added, and the heating continued, with similar addition of alkali, if necessary, until the solution is permanently alkaline, after one hour's heating. The flask is then cooled, and titrated back with N/2 acid. The alkali used for saponification is calculated into acetin (1 c.c. N/2 KOH = 0.0363 Gm. of tri-acetin). (See also *Y.B.*, 1912, 92.)

**Lemon Oil, Detection of Pinene in.** F. C. D o d g e. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1664.) The author utilizes the reaction of pinene nitroso-chloride with aniline, Wallach has shown

that pinene nitroso-chloride, on heating with aniline, regenerates pinene, with formation of amido-azo-benzene :



whereas limonene nitroso-chloride passes, under similar conditions, into a nitrol-anilide :



The reaction thus effects a most satisfactory separation of these terpenes, pinene being isolated as such, in convenient form for further tests if necessary. Unfortunately, the method, like all based on the nitroso-chloride reaction, has only a qualitative value, for the yield of nitroso-chloride depends largely on the rotation of the pinene, being greatest from the inactive variety, and little, if any, being obtainable from pinene of high dextro- or laevo-rotation, and in cases where turpentine oil may have been added to oil of lemon, it is reasonable to suppose that a highly dextro variety would be selected. In many cases, however, positive results may be obtained by operating as follows : 100 c.c. of oil is fractioned very slowly from a three-bulb Ladenburg flask, collecting the first 10 c.c. This distillate is then mixed with 10 c.c. glacial acetic acid and 10 c.c. EtNO<sub>2</sub>, in a freezing-mixture. Three c.c. of 25 per cent. HCl is now slowly added, and the mixture allowed to crystallize for several hours in the cold. The nitroso-chloride is then filtered off, washed with cold methyl alcohol, and dried. The crystals are now mixed with three times their weight of re-distilled, colourless aniline, and eight times their weight of 95 per cent EtOH, in a small flask, and heated gently on the water-bath. A rather vigorous reaction generally ensues. In the absence of pinene, the solution remains light coloured : with pinene, it darkens very noticeably. After half an hour, 25 c.c. water is added, and steam is passed through the flask. Pinene, if present, distils with a little aniline : the latter is dissolved by addition of a little dilute HC<sub>2</sub>H<sub>3</sub>O<sub>2</sub> or HCl, and the pinene remains as insoluble light oil, recognizable by its odour. The limit of sensibility of this method appears to be at about 10 per cent. turpentine in lemon or orange oil. By more careful fractionation of larger amounts of oil, the delicacy of the test could no doubt be increased.

**Lemon Oil, Terpeneless, by Alcoholic Extraction.** J. W. Marden and V. Elliott. (*J. Ind. Eng. Chem.*, 1914, 6, 628.) It is stated that "terpeneless" oil is extracted by shaking out lemon oil with EtOH 45 per cent. Citral is not easily extracted from terpenes by an EtOH solvent of this strength and many shakings-out would be necessary. Alcohol 50 per cent. is more efficient, dissolving at least three times as much citral.

**Lemon-Grass Oil, Cochin, Solubility of, in Alcohol 70 per cent.** (*Bull. Imp. Inst.*, 1914.) Cochin lemon-grass oil marketed in England has been noted to be less soluble, in some instances, in EtOH 70 per cent. than the typical Cochin oil, the solubility of which is 1 : 2 or more of EtOH 70 per cent. Five authentic samples were therefore examined. Three of the five were "insoluble," giving a turbid solution 1 : 5 in alcohol 70 per cent. In order to elucidate the cause of this change of character the kinds of grass used in the distillation were investigated. It was found that two varieties are used, "white-stemmed" grass" and "red-stemmed." Both these grow wild. The oil from the white-stemmed grass was lemon yellow; sp.g. 0.909;  $\alpha_D - 10^\circ 50'$  at  $22^\circ\text{C}$ .; aldehydes by  $\text{NaHSO}_3$  method, 9 per cent.; not soluble 1 : 5 in EtOH 70 per cent.; soluble 1 : 0.8 or more in EtOH 80 per cent., becoming slightly turbid with 1 : 4. The characters of this oil more nearly approach those of citronella oil than of Indian lemon-grass-oil. Specimens of the grass are being obtained for botanical identification. The oil of the "red stemmed" grass had the odour of ordinary lemon-grass oil, the colour was reddish brown: sp.g. 0.925; too dark for optical examination; aldehydes by  $\text{NaHSO}_3$  method, 71.5 per cent.; insoluble 1 : 5 in EtOH 70 per cent.; soluble 1 : 0.8 and more in EtOH 80 per cent., but slightly turbid 1 : 5. When redistilled with steam until 78.7 per cent. of the original oil was obtained, the rectified bright yellow oil was found to be soluble 1 : 2.4 in EtOH 70 per cent. and to contain 81 per cent. of aldehydes. The residue was insoluble. The insolubility of the original oil is therefore probably due to faulty methods of distillation.

**Librocedrus Decurrens, Oil of.** (*Perfum. Record*, 1915, 6, 214.) The oil from the needles of this coniferous tree, which flourishes chiefly in California, has been examined. It has the sp.g. 0.8756,  $\alpha_D - 0^\circ 51'$ , and  $\eta_D 1.47544$ . Soluble 1 : 7 of 90 per cent. EtOH with turbidity. It contains laevo- $\alpha$ -pinene and b-pinene.

The higher boiling fractions had a marked odour, resembling that of carvone, but no semicarbazone could be obtained.

**Lime Oil, Fijian.** (*Bull. Imp. Inst.*, 1914, 12, 227.) The pale yellow oil had the characteristic odour of distilled lime oil; sp.g. 0.863;  $\alpha_D + 37^\circ 6'$ .

**Menthol, Solidifying and Melting Point of.** R. Meldrum. (*Chem. News*, 1915, 111, 229.) Menthol solidifies below its normal solidifying-point, due to assuming the colloidal condition previous to crystallizing. The solidifying-point varies as the colloidal condition varies. Its melting-point is constant at  $42^\circ\text{C}$ ., but solidifying-point varies by  $3.3^\circ\text{C}$ ., when determined by good methods.

**Mustard Oil in Cruciferous Plants.** — Blankma (*Pharm. Weekbl.*; *Drugg. Circ.*, 1915, 59, 167.) The presence of glucosides in various cruciferous plants which on hydrolysis yield mustard oil is reported. The presence of the glucosides was established by various methods such as the odour of the plant, the behaviour of the caterpillars of the common white butterfly, which are said to feed only on plants containing glucosides yielding mustard oil, the action on the growth of the mould, *Saccharomyces mycoderma*, and the phenylhydrazin reaction. Thus butyl iso-thiocyanate was found to be present in *Cochlearia officinalis*, *C. danica*, and *Cardamine pratensis*. *Tropæolum majus* contained benzyl iso-thiocyanate, while *Lepidium campestre*, *Nasturtium amphibicum* and *Draba verna* furnished mustard oils, the chemical constitution of which is still under investigation. *Capsella bursa-pastoris* does not contain mustard oil but a mercaptan.

**Nepeta nepetella, Essential Oil of.** (Roure Bertrand fils' Report, *J.S.C.I.*) The oil distilled from plants gathered in September, after flowering was a yellow viscous liquid having the sp.g. at  $20^\circ\text{C}$ ., 1.03984,  $\alpha_D + 15^\circ 12'$ ; acid value, 45.5; ester value, 245.7; soluble in 2 vols. of EtOH 70 per cent.; it gives no coloration with  $\text{Fe}_2\text{Cl}_6$ . It contains a small amount of aldehyde or ketone.

**Peppermint Oil, Rectification and Examination of.** A. Woehlke. (*Ber. Pharm.*, 1914, 24, 292; *Chem. Abstr.*, 1915, 9, 351.) Peppermint oil may, without material loss, be purified by distillation *in vacuo*, but this procedure possesses no

advantages over those resulting from steam distillation. In the evaluation of peppermint oil, due regard must be given its solubility in 70 per cent. EtOH. The determination of free and combined menthol according to the method of Power and Kleber is very well suited to practical needs and easily carried out. No calculable relationship between the chemical and physical "constants" of peppermint oil can be found.

**Peppermint Oil, Russian.** (*Perfum. Record*, 1914, 5, 315.) A considerable amount of peppermint is cultivated in Little Russia, in the Ukraine, around Poltava. Locally the oil is chiefly used for flavouring tobacco. The output has now outgrown the local demand and amounts to 20,000 lb. of peppermint oil per annum as well as 1,000 lb. of spearmint oil. A specimen of this Russian peppermint oil was found to have the following characters: sp.g. 0.904;  $\alpha_D - 26^\circ$ ; total menthol, 53.8 per cent.; esters as menthyl acetate, 6.0 per cent.; soluble 1:3 in EtOH 70 per cent.; becoming opalescent 1:10. (See also *Y.B.*, 1912, 99.)

**Pinus ponderosa and P. lambertiana, Leaf and Cone Oils of.** A. W. Schorger. (*J. Ind. Eng. Chem.*, 1914, 6, 893.) The needles and twigs of the Western yellow pine, *Pinus ponderosa*, yield from 0.04 to 0.126 per cent. of oil having the sp.g. at  $15^\circ\text{C}$ ., 0.8718 to 0.8849;  $[\eta]_{D_{15}^\circ\text{C.}}$  1.4789 to 1.4838;  $[\alpha]_{D_{20}^\circ\text{C.}}$   $-15.73^\circ$  to  $-19.59^\circ$ ; acid value, 0.67 to 2.36; ester value, 3.88 to 8.10; acetyl value, 24.11 to 35.68. The cones of this tree yield 0.063 per cent. of oil having the sp.g. at  $15^\circ\text{C}$ ., 0.8757;  $(\eta)_{D_{15}^\circ\text{C.}}$  1.4789;  $[\alpha]_{D_{20}^\circ\text{C.}}$   $-11.48^\circ$ ; acid value, 1.27; ester value, 7.20; acetyl value, 22.41. The leaves and twigs of the sugar pine, *Pinus lambertiana*, give from 0.045 to 0.120 per cent. of oil having the sp.g. at  $15^\circ\text{C}$ ., 0.8676 to 0.8738;  $[\eta]_{D_{15}^\circ\text{C.}}$  1.4777 to 1.4794;  $[\alpha]_{D_{20}^\circ\text{C.}}$   $-11.07^\circ$  to  $-16.50^\circ$ ; acid value, 0.68 to 2.38; ester value, 2.22 to 5.91; acetyl value, 23.25 to 32.04. The cones of the sugar pine yielded 0.32 per cent. of oil having the sp.g. at  $15^\circ\text{C}$ ., 0.8692;  $[\eta]_{D_{15}^\circ\text{C.}}$  1.4771;  $[\alpha]_{D_{20}^\circ\text{C.}}$   $-23.18^\circ$ ; acid value, 0.63; ester value, 3.75; acetyl value, 17.04. All contain a trace of furfural except the needle oil of *P. ponderosa*. In all laevo- $\beta$ -pinene is the main constituent. Besides this, all contain laevo- $\alpha$ -pinene, dipentene, and a little ester calculated into bornyl acetate. All but the cone oil of *P. lambertiana* give green oil on fractionation. This oil alone contains laevo-camphene, which in this amounts to 21 per cent.

**Pinus sabiniana, P. contorta, and Abies magnifica, Leaf and Twig Oils from.** W. Schorger. (*J. Ind. Eng. Chem.*, 1915, 7, 24.) *Pinus sabiniana*.—The oil from the leaves and twigs of the Digger Pine had the following characters: sp.g. 0.8517 to 0.8566;  $\eta_{D_{15}}$  1.4670 to 1.4671;  $\alpha_{D_{20}}$   $-20.93^{\circ}$  to  $-38.36^{\circ}$ ; acid value, 1.47 to 2.05; ester value, 6.77 to 11.98; acetyl value, 25.86 to 37.16. The following constituents were isolated in the percentages indicated:  $\eta$ -Heptane, 3; laevo- $\alpha$ -pinene, 58–59; laevo-limonene, 18; bornyl acetate, 3.5; free alcohol (as borneol), 6; with 2 to 3 per cent. of green oil. The yield of oil was from 0.078 to 0.102 per cent. *Pinus contorta*.—The leaves and twigs of the Lodge-pole Pine gave 0.234 per cent. of oil having the sp.g. 0.8690;  $\eta_{D_{15}}$  1.4831;  $\alpha_{D_{20}}$   $-17.84^{\circ}$ ; acid value, 0.9; ester value, 6.02; acetyl value, 32.39. A trace of furfural occurred in the first runnings. The oil contained the following percentages of the constituents named: laevo-camphene, 5 to 6; laevo- $\beta$ -pinene, 49 to 50; laevo- $\alpha$ -pinene, 3; laevo-phellandrene and dipentene, together 19; bornyl ester as acetate, 2; free alcohol as borneol, 7.5; cadinene, 7. *Abies magnifica*.—The oil from the leaves and twigs of the Red Fir was light green in colour and had a pungent unpleasant odour. The yield was 0.154 per cent.; sp.g. 0.8665;  $\eta_{D_{15}}$  1.4861;  $\alpha_D$   $-16.70^{\circ}$ ; acid value, 0.75; ester value, 9.93; acetyl value, 36.22. The oil contained a trace of furfural and the following constituents in the approximate percentages named: laevo- $\beta$ -pinene, 16 to 18; laevo-phellandrene, 52; bornyl ester as acetate, 3.8; free alcohol as borneol, 7.5; green oil, 13.

**Prostanthera cineolifera, Essential Oil of.** R. T. Baker and H. G. Smith. (*J. Proc. Roy. Soc. N.S. Wales*, 46, 103; *Chem. Abstr. Amer. Chem. Soc.*, 1914, 8, 2411). *Prostanthera cineolifera* is shown to contain cineol as its principal oil constituent; also cymene and probably geraniol. Thymol and carvacrol were also present. The aldehyde prepared from the oil proved to be cuminaldehyde.

**Ramona stachyoides, Oil of.** C. E. Burke and C. C. Scallione. (*J. Ind. Eng. Chem.*, 1914, 6, 804.) Black sage, *Ramona stachyoides*, grows extensively in California. A gathering of several hundred pounds made in February yielded 0.90 per cent. of oil having the following characters: sp.g. 0.8979; specific rotation, 24.4;  $\eta_D$  1.4729; insoluble in EtOH 70 per cent.; acid value, 2.2; ester value, 1.6; separating no solid



matter when cooled to  $-20^{\circ}\text{C}$ . It had the following percentage composition : Pinene, 6 ; cineol, 30 ; dipentene, terpinene, etc., 25 ; thujone, 8 ; camphor, 8 ; resinoid matter, 5. These figures differ somewhat from those obtained in a previous analysis published by the Bureau of Plant-Industry of an oil derived from material gathered in the month of April, when the plants were in bloom. The yield then obtained was 0.75 per cent. The oil contained 62.5 per cent. of camphor, part of which crystallized out when the liquid was cooled to  $-20^{\circ}\text{C}$ . It also gave 22.5 per cent. of cineol.

**Santolina chamaecyparissus, Oil of.** L. Francesconi and N. Granata. (*Gazz. Chim. Ital.*, 1914, **44**, 1, 150 ; *Chem. Abstr.*, 1915, **9**, 620.) The oil contains two unsaturated ketones  $\alpha$ - and  $\beta$ -santolenone  $\text{C}_{10}\text{H}_{16}\text{O}$  ; and a third saturated ketone  $\gamma$   $\text{C}_{10}\text{H}_{16}\text{O}$ , probably a camphor-like substance ; this is laevorotatory. All three combine with  $\text{NaHSO}_3$  with difficulty and fail to give crystalline Br products. Thujone was not detected in the specimen of oil examined. A terpene-like liquid with a pleasant odour was also found.

**Solidago memorialis Oil.** E. B. Miller and M. H. Eskew. (*J. Amer. Chem. Soc.*, 1914, **36**, 2538.) The fresh herb of the American ragweed, collected in October, gave 0.322 per cent. of oil ; sp.g. 0.8532 at  $\frac{25}{25}^{\circ}$  ;  $\alpha_D$   $-16.17^{\circ}$  ;  $\eta_{D18}$  1.47397 ; saponification value, 5.6 ; acetyl value, 9.4 ; soluble 1 : 4 in EtOH 90 per cent., 1 : 24 in EtOH 70 per cent. The chief constituent is  $\alpha$ -pinene. Salicylic and acetic acids are also present, and at least one alcohol, probably borneol, free and as esters.

**Solidago rugosa and S. odora, Essential Oils of.** E. R. Miller and J. M. Moseley. (*J. Amer. Chem. Soc.*, 1915, **37**, 1285.) The flowering plants of the golden rod, *Solidago rugosa*, collected in Alabama yielded 0.4 per cent. of light yellow oil ; sp.g. 0.8620 at  $\frac{25}{25}^{\circ}\text{C}$ . ;  $\alpha_D$   $-12.8^{\circ}$  ;  $\eta_D$  1.4813 ; saponification value, 4.22 ; acetyl value, 10.97. It consisted mainly of terpenes of which at least three were present : one of these was  $\alpha$ -pinene. A small amount of an ester equivalent to 1.47 per cent. of bornyl acetate, and a free alcohol equivalent to 1.67 per cent. of borneol, were also present. *Solidago odora*, the sweet-scented golden rod, or Blue Mountain tea, was formerly official in the U.S.P., being used as a carminative and flavouring agent. The plant is also employed in domestic medicine in the U.S.A. A small

amount of the oil is occasionally met with in commerce, but the botanical source of this is doubtful. A series of ten distillations made by the authors from the flowering herb during September and October gave from 0.64 to 1.53 per cent. of oil. The bulked oil of nine of these, from wild plants, had the sp.g. 0.9310 at  $\frac{25}{25}$ ° C.;  $\alpha_D + 13.72^\circ$ ;  $\eta_{D25} 1.5065$ ; saponification value, 7.9; acid value, 0.63. The oil was sweetish in taste and had the odour of anise, with a suggestion of saffrol. The main constituent was methyl chavicol, of which 75 per cent. of the oil consisted; it contained as well terpenes, 10 to 15 per cent.; esters about 3 per cent., and free alcohol about 3 per cent., calculated as borneol. Small quantities of volatile fatty acids and a non-volatile acid were indicated.

**Star Anise Oil in 1915.** (*Perfum. Record*, 1915, 6, 105.) The quality of star anise oil imported during 1915 to date (April) shows a marked improvement over the imports during the previous year, as indicated by sp.g., m.p., and congealing point. In 1914 the sp.g. ranged from 0.979 to 0.983 at 20/15°C.; m.p. from 16.7 to 17.2; congealing point, from 14.7° to 15.2°C. In 1915 the sp.g. has been from 0.982 to 0.987 at 20/15°C.; m.p., 17.0 to 18.8; congealing point, 15° to 16.8°C. The sample with the highest sp.g. and congealing point was slightly dextrogyre. All the others were slightly laevogyre, or optically inactive.

**Sweet Birch Oil and Methyl Salicylate, New Colour Reactions to Distinguish.** G. N. Watson and L. E. Sayre. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1658.) An excess of  $H_2SO_4$  gives, with the natural oil, a dark red colour. The reagent produces no colour with the synthetic oil. With oil of birch, a yellow or light shade of red is produced. For a confirmatory test, an EtOH solution of heliotropin and sulphuric acid makes a good reagent. To a few drops of the oil add 2 c.c. of concentrated  $H_2SO_4$  and two drops of a saturated alcoholic solution of heliotropin. This reagent gives, with the natural oil, a crimson colour, changing to deep violet upon dilution with alcohol. Oil of birch gives practically the same colour, but not so pronounced. With the synthetic oil the reagent produces a bright yellow colour, due, however, to the action of the acid on the heliotropin and not to any action on the oil. A second confirmatory reagent, and one superior to heliotropin, since it differentiates the oil of wintergreen and oil of birch, is an aqueous solu-

tion of chloral hydrate and  $\text{H}_2\text{SO}_4$ . To 1 c.c. of the oil in a test tube add 2 c.c. of concentrated  $\text{H}_2\text{SO}_4$ , then 1 c.c. of a saturated aqueous solution of chloral hydrate. With the natural oil a green colour develops, a dark green oil-layer above a lighter green aqueous zone. The addition of 2 or 3 c.c. of water aids in bringing out these shades. Oil of birch gives a deep violet oil-layer. The synthetic oil produces no colour except after long standing, when a faint violet tint may develop. (See also *Y.B.*, 1914, 60.)

**Terpenes, Action of Mercuric Acetate Reagent with.** L. Balbiano. (*Ber.*, 1915, 48, 394; *Chem. Abstr.*, 1915, 9, 1486.) By means of saturated  $\text{Hg}(\text{OAc})_2$  paraffins, aromatic hydrocarbons, cycloparaffins, olefines and terpenes can be detected and determined in mixtures of hydrocarbons. Purest pinene, prepared by Wallach's method from the nitroso-chloride, when boiled 2-4 hours under a reflux condenser with the required amount of  $\text{Hg}(\text{OAc})_2$  in four parts of water, is completely decomposed; a large part resinifies, but about 50 per cent. is converted into dihydroxypinene (isolated as the semicarbazone); no appreciable amount of cymene is formed. Petroleum ether and pinane are not attacked and a large part is recovered when mixtures with pinene are treated as above with  $\text{Hg}(\text{OAc})_2$ . Camphene boiled two hours with aqueous  $\text{Hg}(\text{OAc})_2$  in the presence of  $\text{AcOH}$  gives a precipitate of the compound  $\text{C}_{10}\text{H}_{16}\text{OHg}(\text{OAc})_2$  (a) which regenerates camphene with  $\text{Zn}$  and  $\text{HCl}$  on the water-bath; if  $\text{C}_6\text{H}_6$  or pinane are also present, they are largely recovered unchanged. The following is an example of a mixture analyzed by this method: 5 c.c. pinene, 2 c.c. cymene, 2 c.c. pinane and 1 Gm. camphene are boiled  $2\frac{1}{2}$  hours with 33 Gm.  $\text{Hg}(\text{OAc})_2$  in 132 c.c.  $\text{H}_2\text{O}$ ; from the steam distillate of the reaction mixture there separates 3.5 c.c. of an oily mixture of cymene and pinane which no longer reacts with  $\text{Hg}(\text{OAc})_2$ , showing that the reaction is complete. From the precipitate of  $\text{HgOAc}$  and (a) there is obtained, by treatment with  $\text{Zn}$  and  $\text{HCl}$  and subsequent steam distillation, about 0.6 Gm. camphene. The aqueous solution is concentrated on the water-bath, the residue dissolved in  $\text{Et}_2\text{O}$ , the solution washed with  $\text{H}_2\text{O}$  and concentrated; there remains a red-yellow oil which gives 0.8 Gm. of dihydroxypinene semicarbazone.

**Terpenyl Acetate, Detection of, as an Adulterant of Essential Oils.** C. L. Barillet and R. Berthelé. (*Bull. Soc.*

*Chim.*, 1915, 17, 20.) Terpenyl acetate (and presumably other esters of terpenol) is saponified much less quickly in the cold with alcoholic KOH than linalyl or geranyl acetates. Thus, linalyl acetate had the saponification value of 85.5 with alcoholic N/2 KOH after boiling for 3 hours, and 80.3 after standing in the cold for 24 hours. Under like conditions the "hot" saponification value of terpenyl acetate was 92.1 and the "cold" value only 32.6. Mixtures of the two esters gave corresponding results. Consequently it is easy to detect the addition of terpenyl acetate for the purpose of giving fictitiously high ester values to certain essential oils, such as bergamot oil. Two saponifications are made with alcoholic N/2 KOH, one by boiling for 3 hours, the other by standing in the cold for 24 hours. Any great divergence in the two results will indicate an added ester, probably a terpenyl ester.

**Thymol, Melting and Solidifying Points of.** R. Meldrum. (*Chem. News*, 1915, 111, 193.) Various figures ranging from 44° to 50°C. have been given as the m.p. of thymol. The author has investigated the matter and finds that these divergent results are due to the various conditions under which the experiments are performed, to the presence of moisture, and to the ease with which thymol enters into superfusion, and remains so for a time. The true melting point determined by the thermometer bulb method is 48.7 to 49.2°C. The solidifying point varies between 48.2 and 49.2°C. The higher figure may be accepted as it approaches that of the m.p.

**Vetiver Oil, Fijian.** (*Bull. Imp. Inst.*, 1914, 12, 225.) The oil was yellowish-brown in colour, becoming dark green on exposure to air. Sp.g. 1.018; solubility in EtOH, 80 per cent. 1:1.3, becoming turbid with 1:4; saponification value, 47. It will be seen that this oil differs materially from the sample of Fijian vetiver oil examined in 1912 (*Y.B.*, 1912, 108). It resembles the heavy oil distilled in Europe, rather than the light oil of Réunion. (See also *Y.B.*, 1913, 96.)

**Ylang-ylang Oil from Seychelles and Mauritius.** (*Bull. Imp. Inst.*, 1914, 12, 228.) *Seychelles oil.*—The oil had the sp.g. 0.9567;  $\alpha_D$   $-28^{\circ}5'$  at 20°C.; acid value, 3.3; ester value, 126; acetyl value, 181; solubility in EtOH 90 per cent.; clear in less than 1:0.8, turbid with 1:0.8 and more. These characters agree with those of first grade Manila oil. This oil

is superior to that previously received from the same source. *Mauritius oil*.—The oil was yellow and turbid. Sp.g. 0.9883;  $a_D - 30^\circ$  at  $20^\circ\text{C}$ .; acid value, 7; ester value, 173; acetyl value, 180; solubility in EtOH 90 per cent.; clear from 1:0.1 to 1:2.5, turbid with more. The ester value is abnormally high.

## FATS, FIXED OILS AND WAXES

**Beeswax, Bleaching of.** K. Heinz. (*Seifensied. Zeit.*, 1913, 40, 1140, 1169, 1192; *J.S.C.I.*, 1915, 33, 1098.) Complete bleaching of beeswax can only be effected by sunlight, and all chemical methods require supplementing by sun-bleaching. Moreover, it is frequently impossible to remove all traces of chemicals from the product. Natural bleaching is effected by repeated exposures, the wax being melted with water containing  $\text{H}_2\text{SO}_4$  between each exposure. Of chemical agents, oil of turpentine has the drawback of imparting an unpleasant odour to the wax. It is preferable to add to the water, used to prevent overheating of the wax by the sun,  $\text{KClO}_3$ , sodium hypochlorite, or  $\text{H}_2\text{O}_2$ . Another method is to boil 100 kilos. of the melted wax with a solution of 3 kilos. of  $\text{KClO}_3$  in 10 litres of water acidified with  $\text{H}_2\text{SO}_4$ , and after removal of the chemicals, to finish the bleaching in the sun.  $\text{KMnO}_4$  is not effective as a bleaching agent. One of the best methods is to boil the wax with a solution of  $\text{K}_2\text{Cr}_2\text{O}_7$  and  $\text{H}_2\text{SO}_4$ , and to remove the chromic oxide by washing with water containing lactic acid or  $\text{H}_2\text{SO}_4$ . Ozone has not proved very effective. Bleaching with fuller's earth, Florida earth, and the like, has the drawback that the powder retains a considerable amount of wax, which can only be recovered by extraction.

**Beeswax, Detection of Small Quantities of Paraffin in.** M. S. Salamon and W. M. Seabers. (*J.S.C.I.*, 1915, 34, 461.) Advantage is taken of the less solubility of paraffins in hot EtOH than that of beeswax. When saponification is conducted in the usual manner, by boiling with N/2 alcoholic KOH, the temperature at which turbidity appears in the case of pure European beeswax is constant between  $59.5^\circ$  and  $60^\circ\text{C}$ . For waxes of the East Indian type it ranges from  $56^\circ$  to  $57^\circ\text{C}$ . In the presence of 5 per cent. of paraffin, these temperatures are considerably modified. The test is applied thus. One Gm. of wax is saponified over the flame for 1 hour with 10 c.c. of N/2 alcoholic KOH

and 10 c.c. of industrial EtOH. The flask is taken off the flame and a thermometer inserted, and the liquid stirred continuously until at a certain temperature the solution becomes cloudy. The point is very sharp and constant. In the case of pure waxes, the cloudiness is followed by immediate precipitation of large flocks; with adulterated samples, however, the clouding is gradual and flocculation does not occur until a lower temperature is reached. The presence of carnauba, stearin, insect, or Japan wax, does not appear to interfere appreciably with this point, but the presence of as little as 5 per cent. of paraffin wax raises the point considerably. In the case of waxes of the East Indian type, 5 per cent. of paraffin will raise the point from 56°C., the figure for pure waxes, to 61°–62°, and 10 per cent. raises it to 69°–90°C. With waxes of the European type, the temperature of clouding rises from 60°C. for pure waxes, to 63°–64°C. for 5 per cent. paraffin, and to 74°–75°C. for 10 per cent. paraffin.

The following figures show the effect of the addition of 5 per cent. and 10 per cent. of paraffin wax to some of the usual varieties met with in commerce.

#### WAXES OF THE EUROPEAN TYPE.

	Original Pt.	5 per cent. Paraffin.	10 per cent. Paraffin.
Benguella beeswax . .	60°	64°	74·5°
Spanish beeswax . .	60°	63°	73·5°
Morocco beeswax . .	60°	65°	75°
East African beeswax .	60°	64°	74°
English beeswax . .	60°	66°	75°
West African beeswax	60°	64°	75°
Abyssinian beeswax .	60°	64·5°	75°

#### WAXES OF THE EAST INDIAN TYPE.

	Original Pt.	5 per cent. Paraffin.	10 per cent. Paraffin
Chinese beeswax	56°	62°	70°
Calcutta beeswax	56°	62°	70°

The paraffin wax used varied in melting point from 56°C. to 60°C.; for paraffin of very low melting point such as 42°C. the

clouding point is a little lower, usually about 63°C. for 5 per cent., but the presence of such a paraffin so affects the melting point and the other physical characters that its presence is usually easily detected, and moreover it is very rare for a low-melting paraffin to be used as an adulterant, the adulterators usually selecting one with a melting-point nearer that of beeswax itself. The following figures of three commercial samples recently examined may be of interest.

No.	Acid Value.	Ester Value.	Melting Point.	Clouding Point.
1	18.5	68	62°C.	Not clear
2	17.9	71.7	63°C.	64°C.
3	17.9	70	63°C.	67°C.

It will be noticed that the ester and acid values of all of them are somewhat low, and this, coupled with the clouding point, leaves little doubt that they were all adulterated with from 5–15 per cent. of paraffin. It also may be noticed that although these samples were adulterated, yet the figures are well within the limits laid down by the new B.P.

In the discussion which followed W. F. Reid stated that many samples of genuine wax contained a good deal of unsaponifiable matter. Propolis was often taken into solution when rendering wax from the comb. Old, pollen-clogged combs yielded with difficulty a wax which was quite different from that obtained from virgin comb. The impression that "foundations" for artificial combs was sometimes made of ceresin or paraffin was incorrect, at any rate in this country. If such material were given to bees, they would tear it down. The wax made by Indian or African bees, of a different species from the English or European bee, had a lower melting point. If it were used for foundations for British bees, they tore it down. The odour of foreign bees was hateful to our species, and they rejected their wax.

**Ceresin Wax Adulterated with Rosin.** E. J. Parry. (*Chem. & Drugg.*, 1914, 85, 376.) The scarcity of European petroleum products has led to gross adulteration of some grades of paraffin waxes. Samples have been met with grossly adulterated with rosin. These were sticky, of indefinite m.p. and tasted of rosin. They gave well-marked Storch-Moraivski reaction. The acid

value ranged from 48 to 68 ; saponification value from 51 to 72·5 ; and iodine value from 40 to 56.

**Croton Elliottianus Seeds, Fixed Oil from.** (*Bull. Imp. Inst.*, 1915, 13, 39 ; also J. T. Cash and W. J. Dilling, *Journ. Pharmacol. Exp. Therap.*, 1914, 6, 235.) The oil from the seeds of this East African Euphorbiaceous tree was first extracted at the Imperial Institute in 1907, which differed from the oil of *Croton Tiglium* in not having vesicating properties when applied to the skin. A second consignment of the seeds has yielded 57·4 per cent. of dark yellow, almost tasteless oil ; sp.g. 0·927 ; acid value, 3·6 ; iodine value, 147 ; saponification value, 191·6 ; titre test, about 14° ; Hehner value, 94·8. Physiological experiments show that towards man the seed of *C. Elliottianus* is laxative when taken in small doses (0·1 to 0·2 Gm.), but rapidly purgative (bordering on drastic) in larger amount (0·4 Gm.) The oil exerts a similar action, but it is less irritant than the seeds and its action is much more uniform. The authors express the opinion that the relatively non-irritant action of *C. Elliottianus* oil, its certain effect, whether as a laxative in 8 to 10 hours or in larger dose as a speedy purgative, its high potency in relationship to the small bulk, indicate it as a body which would be of considerable value as an addition to purgative remedies, for some of the more drastic and irritant of which it would prove a safe and effective substitute.

**Grape-Seed Oil.** G. Dell'Acqua. (*Annali Chim. Applic.*, 1914, 2, 295 ; *Analyst*, 1915, 40, 54.) The increasing cost of olive oil has led to the introduction of grape-seed oil as a commercial product. A pure extracted sample had a greenish-yellow colour similar to that of inferior olive oil, and gave the following values : Sp.g. at 15°C., 0·9226 ; Valenta test, 83°C. ; refractometer reading (Zeiss), 78·8 at 15°C., 62·9 at 40°C. ; iodine value, 140·25 ; acetyl value, 17·84 ; unsaponifiable matter, 0·32 per cent. *Fatty Acids.*—Sp.g. at 25°C., 0·8988 ; m.p., 25° to 28·5°C. ; solidifying point, 21° to 18°C. ; refractometer reading, 62·8 at 20°C. ; 51·8 at 40°C. ; and iodine value, 141. The oil thus resembled Soya-bean oil in many of its constants, and, like that oil, gave a lemon-yellow emulsion in the uranium nitrate test. The oils could be distinguished, however, by heating 10 c.c. with 3 c.c. of an Et<sub>2</sub>O 2 per cent. solution of uranium nitrate for 2 minutes in a boiling NaCl bath (102°C.).



Soya-bean oil assumes an olive-green colour changing to garnet red within 20 minutes, while grape-seed oil becomes yellowish-green in 2 minutes and golden-yellow within 20 minutes. Hauche-corne's nitric acid test will also distinguish between the two oils. Soya-bean oil heated for 10 minutes in water at 60°C. with nitric acid gives an orange-brown colour changing to chocolate-brown, while grape-seed oil assumes an orange-brown colour changing to reddish-orange.

**Hedera helix Seeds, Iso-oleic Acid in.** F. C. P a l a z z o and A. T a m b u r e l l o. (*Atti Accad. Lincei*, 1914, 23 [2], 362; *Chem. Abstr.*, 1915, 9, 1476.) The non-saturated fatty acid occurring in the seeds of ivy berries, which was at first thought to be brucic acid, is now found to be iso-oleic acid. This is probably identical with petroselinic acid from parsley seeds. The air-dry seeds contain from 30 to 32 per cent. of total fat.

**Mowrah Fat, Detection of, by the Optical Rotation of its Unsaponifiable Constituents.** P. B e r y and J. A n g e r h a u s e n. (*Zeits. Untersuch. Nahr. Genussm.*, 27, 723; *Chem. Abstr. Amer. Chem. Soc.*, 1914, 8, 3207.) The unsaponifiable portion of mowrah fat contains a dextrorotatory substance having the  $[\alpha]_D + 34^\circ$ . This may serve to detect the admixture of mowrah fat with other animal and vegetable fats. For instance the unsaponifiable residue of lard is laevorotatory,  $[\alpha]_D - 19.6^\circ$ . This is due to cholesterol. After separating this by means of EtOH and digitonin, the residue is optically inactive. In order to detect mowrah fat in admixture with other fats, the cholesterol is removed as indicated, and the optical rotation of the residue in  $\text{CHCl}_3$  is determined. The  $[\alpha]_D$  of the cholesterol-free unsaponifiable matter from an admixture of 10 per cent. of mowrah fat with lard was  $+34.9^\circ$  in  $\text{CHCl}_3$  solution.

**Oils and Fats, New or Little Known.** E. R. B o l t o n and E n i d M. J e s s o n. (*Analyst*, 1915, 40, 3.) A number of oleaginous fruits or seeds are described as possible sources of oils. The yields of the oils and their chemical and physical characters are given in tabular form. The oils include those from the fruits of the following plants. *Balanites maurhamii* N.O. *Simarubaceae* from Portuguese East Africa; yielding mandure oil. The sticky pulp contains an olive-green oil with an overpowering odour of butyric acid. The kernel oil has only a slight odour. *Calophyllum tomentosum* N.O. *Guttiferae*

from India gives a pale yellow fat of the consistence of vaseline and has an unpleasant odour. *C. tomentosum*, which is widely distributed and yields a brownish olive-green oil with a marked odour recalling aniseed or fenugreek. It has been examined previously. *Melia azadirachta*, the Indian neem tree, the oil of which has been frequently examined. The fruit and oil have a strong garlic-like smell. *Fevillea cordifolia* N.O. *Cucurbitaceae* of Tropical America and West Indies, known as the Jamaica "Antidote Cacao," yields a pale cream-coloured plastic fat with a bitter taste and a slightly unpleasant odour. Its  $\eta_D$  is exceptionally high. *Telfairia pedata* N.O. *Cucurbitaceae*, from Tropical Africa fruits, yield an oil known as Koeme oil, which is pale yellow with a reddish or greenish fluorescence, and having a slightly bitter taste; although both oil and seeds are eaten by natives, the former may be poisonous if the pulp of the fruit is expressed with the seeds. *Canarium luzonicum*, of the Philippine Islands, yields elemi, known locally as Brea blanca. The seeds have a very hard thick shell which is difficult to remove. The kernel contains a sweet bland oil resembling sweet almond oil. *Canarium commune* seeds yield a similar oil, known as Javan almond oil. *Schleichera trijuga*, from India and Malay, yields nuts known as Kusambi or Pacca nuts, and the tree is called the Ceylon oak. The oil is known as Kon or Kusum oil and is said to be the original Macassar oil. *Sterculia fetida* is widely distributed in the tropics, where the seeds are known as Java olives. The kernels yield a pale yellow viscous oil without marked odour or taste. It solidifies on being heated to 250°C. *Anacardium occidentale*, from Central America, furnishes the cashew nuts or promotion nuts, the kernels of which are largely used by confectioners. They yield a pale yellow oil. The black pericarp yields a very dark oil which has the vesicant and irritant properties of the fruit. Cashew nuts are always slightly burnt, since torrefication is employed to remove the outer portions. *Buchania latifolia*, from India and Burma, yields "Peru palm kernels." These are used by the natives in confectionery, the kernels resembling pistachio nuts in flavour. They yield an edible oil, known as "Chironjii oil." *Enocarpus distichus*, from South America, contain a brownish-green fat of lard-like consistence. "Marquaqua nuts," of undetermined botanical source, from Portuguese East Africa, yield a golden yellow, somewhat viscous oil without marked taste or odour.

**Osteophloeum platyspermum Seeds, Fat from.** E. M. JESSON. (*Kew Bulletin*, 1914 [9], 333; *J.S.C.I.*, 1915, 34, 499.) A shipment of these seeds from a tree indigenous to N.W. Brazil was recently received at Liverpool. On extracting with petroleum ether, a white, crystalline fat was obtained. The almost odourless kernels gave 55.2 per cent. of fat, with m.p. 43°C., and solidifying point 39°C.; iodine value (Wijs), 6.3 per cent.; saponification value, 240.2; refractometer reading at 40°C., 36.9. The oil contained 5.3 per cent. of free fatty acids (as oleic acid).

**Pentadesma Kerstingii Fat.** H. WAGNER, J. MUESMANN, and J. B. LAMPART. (*Z. Unters. Nahr. Genussm.*, 1914, 28, 244-249; *J.S.C.I.*, 1915, 34, 366.) The seeds of *Pentadesma kerstingii*, when extracted with Et<sub>2</sub>O, yield 41.5 per cent. of a fat similar in appearance to butter fat but somewhat harder, having the following characters: M.p. 38°-39°C.; solidifying point, 29.2°C.; refractometer reading, 45-46 at 40°C.; acid value, 12.4; Reichert-Meissl value, 0.22; Polenske value, 0.4; saponification value, 192; iodine value, 45.9; unsaponifiable matter soluble in ether, 0.6 per cent. The fat gives no reaction with the reagents of Baudouin, Soltsien, and Halphen; with Bellier's reagent it gives a blue-violet coloration changing soon to wine-red.

**Prunus domestica, The Oil and Amygdalin Content of the Seed Kernels of.** G. KASSNER and K. ECKELMANN. (*Arch. Pharm.*, 1914, 252, 402; *J.S.C.I.*, 1915, 34, 668.) Plum-stone kernels from trees grown on well manured soil yielded 42.92 per cent. of pale yellow, fatty oil of mild taste similar to almond oil, having sp.g. at 15°C., 0.916; acid value, 1.44; ester value, 186.66; iodine value, 104. The kernels contained 1.82 per cent. of amygdalin.

**Spermaceti, M.p. of.** R. MELDRUM. (*Chem. News*, 1915, 111, 37.) A series of experiments by the various methods of determining m.p. and congealing point are thus summarized: Solidifying point by Dalican's method, 45.75-45.93°C.; solidifying point in small bore tube, 45.75-45.93°C.; solidifying point by slow cooling, 45.75-45.93°C.; solidifying point by stirring, 45.75°C.; solidifying point, opacity method, 45.8-46.0°C.; solidifying point, capillary tube, 44.7-45.0°C.; melting point, thermometer bulb method, 45.3-45.5°C.; melting point, open

capillary tube, 45.1–45.4°C.; melting point, closed capillary tube, 45.9–46.0°C.; melting point, opacity method, 46.0–46.4°C.

**Strophanthus Seeds, Fixed Oil of.** H. Matthes and L. Rath. (*Arch. Pharm.*, 1914, 252, 683; *J.S.C.I.*) The oil from *Strophanthus kombé* seeds contains 21 per cent. of solid saturated and 73 per cent. of unsaturated fatty acids. The saturated acids contain 30 per cent. of stearic acid and 70 per cent. of palmitic acid. The liquid fatty acids are a mixture of 80 per cent. of oleic acid and 20 per cent. of linolic acid. *Strophanthus* seed oil only contains one phytosterol, sitosterol (m.p. 137°; acetate, m.p. 127°–128°C.), and does not contain arachidic acid.

**Strophanthus Seed Oil, Unsaponifiable Constituents of.** A. Heiduschka and R. Wallenreuter. (*Arch. Pharm.*, 1914, 252.) *Strophanthus* seed oil contains 1.12 per cent. of unsaponifiable constituents of which 0.504 per cent. is sitosterol.

**Trichilia Seeds from Nigeria, Fat of.** P. Ammann and J. Vuillet. (*L'Agron. Coloniale*, 1914, 2, 34; *J.S.C.I.*, 1915, 34, 288.) Seeds of *Trichilia emetica* from E. Africa have for a long time been imported into France under the name of “ma-furaires”; they contain much fat suitable for soap and candle manufacture. The seeds of various other species of *Trichilia* from French W. Africa have been analyzed, and the best, which were long and orange-coloured, and consisted of 58.2 per cent. of kernels and 41.8 per cent. of shells, contained 43.7 per cent. of fat in the kernels and 51.9 per cent. in the shells. These fats were of a light brown colour, and contained respectively 90.3 per cent. and 92.0 per cent. of fatty acids, of which the melting points were 51.5°C. and 44°C., and the solidifying points 47.2°C. and 40.5°C.; the glycerides solidified at 15°–16°C. and about 13°C. The acidity (as oleic acid) was 2.82 and 3.05 per cent. respectively.

## GLUCOSIDES, SUGARS, AND FERMENTS

**Alpha Glucosidase, Influence of Acetic Acid on.** E. Bourquelot and A. Aubry. (*Comptes rend.*, 1915, 160, 742.) Alpha glucosidase is very sensitive to the presence of free acids. A mere trace of free acetic acid absolutely destroys both its synthetizing and hydrolizing power. This proves that these two functions pertain to one and the same enzyme.

**Anhydrogitalin and a By-product of Digitoxin Manufacture.**  
H. Kiliani. (*Ber.*, 1915, 48, 334-49; *Chem. Abstr.*, 1915, 9, 1336.) The author's crude anhydrogitalin was purified as follows: 10.5 Gm. were allowed to stand, with frequent shaking, in 20 parts of a mixture of equal volumes of MeOH and  $\text{CHCl}_3$ , which left 2.65 Gm. (vacuum-dried) undissolved (*a*); the filtrate, slowly treated with 450 Gm. of  $\text{Et}_2\text{O}$  and allowed to stand 4 days in sealed vessels, gave a granular precipitate which, after washing with  $\text{Et}_2\text{O}$ , drying *in vacuo*, extracting with 4 parts MeOH and again drying, yielded 3.46 Gm. more of (*a*), which is pure anhydrogitalin; it remains almost pure white up to  $250^\circ$ , then sinters gradually and melts at  $255^\circ$ ; its composition is  $\text{C}_{33}\text{H}_{52}\text{O}_{12}$  (Kraft gives  $\text{C}_{28}\text{H}_{46}\text{O}_9$ ); 4.57 Gm. heated for 20 minutes on the water-bath in ten parts of 0.5 per cent. "cleavage acid" (1 c.c. HCl (sp.g. 1.19) in 100 c.c. of 50 per cent. EtOH), then treated with 10 parts water, allowed to stand 24 hours, filtered, washed with water and dried *in vacuo*, gave 1.7934 Gm. of *genin*,  $\text{C}_{21}\text{H}_{30}\text{O}_5$ , wartlets from 15 parts of boiling 96 per cent. EtOH, behaving towards  $\text{Fe-AcOH-H}_2\text{SO}_4$  like Kraft's product but melts  $200^\circ$  (instead of  $216-9^\circ$ ). These facts confirm the view that the anhydrogitalin pre-exists in the original "gitalin" and is not formed by subsequent dehydration.

Samples from Merck, labelled "By-product in the preparation of digitoxin, originally soluble in  $\text{CHCl}_3$  but having become insoluble in the course of manufacture," were allowed to stand 2 days in 4 parts MeOH, with frequent shaking, draining and washing; this removed about 6 per cent., remaining as a deep green, smeary residue, difficult to dry, when the alcohol was evaporated off. The part insoluble in MeOH retains the last traces of the green colouring matter with extraordinary firmness and is extremely difficultly soluble in all the usual solvents, even boiling EtOH. In  $\text{C}_6\text{H}_5\text{N}$  it is soluble in about 7 parts, but  $\text{H}_2\text{O}$  precipitates the colouring matter along with it, and even with much  $\text{H}_2\text{O}$  the precipitation is far from complete; only when the  $\text{C}_6\text{H}_5\text{N}$  is neutralized with an acid is the precipitation complete. Accordingly 20 Gm. of the product were treated with the "cleavage acid" as above for (*a*); this gives 42-5 per cent. of *genin* (somewhat more is obtained by extracting the filtrate and wash waters with  $\text{CHCl}_3$ , drying with  $\text{Na}_2\text{SO}_4$ , evaporating and freeing the residue of soluble products by short warming with  $\text{H}_2\text{O}$ ); this is recrystallized from 4, then from 20 parts of 50 per cent. AcOH with charcoal; this

removes the green colouring matter, but a final crystallization from 12 parts of 95 per cent. EtOH is essential to remove the AcOH. It forms leaflets, m.p. 205–6°, neutral to litmus, reacts like (a) with Fe-AcOH-H<sub>2</sub>SO<sub>4</sub> and probably has the composition C<sub>22</sub>H<sub>32</sub>O<sub>6</sub>; heated 45 minutes in a pressure bottle on the water-bath in 10 parts 50 per cent. EtOH with 1 mol. of N/0.5 NaOH (calculated on the basis of C<sub>22</sub>H<sub>32</sub>O<sub>6</sub>) it yields a clear, faintly yellow solution no longer reacting with phenolphthalein; while if 1 mol. NaOH is taken on the basis of C<sub>19</sub>H<sub>28</sub>O<sub>5</sub>, the phenolphthalein reaction persists after 1.5 hours' heating. On cooling the former solution, needles (7–8 per cent. of the genin used) separated; they contained about 2.5 per cent. Na, began to sinter 225°, then swelled and turned brown without melting. The filtrate, after removal of the EtOH at 35°, gives with HCl an amorphous precipitate of the same composition and neutralizing the same amount of NaOH, on warming, as the original genin but differing from it in being faintly acid on moistening with 50 per cent. EtOH and in not crystallizing. Apparently, therefore, NaOH acting on genin, opens up a lactone union and on acidification a different lactone is reformed. The alkaline solution obtained in the titration of genin, when diluted 1 : 50, gives only an opalescence with BaCl<sub>2</sub> and CaCl<sub>2</sub> (1 : 10), a slight turbidity with 1 : 2 MgCl<sub>2</sub> and voluminous precipitates with ZnSO<sub>4</sub> and CuSO<sub>4</sub>. The Na salt, after removal of the EtOH, is readily attacked by KMnO<sub>4</sub> (4 atoms O per mol. (b)). In an attempt to prepare a genin benzoate, 0.7125 Gm. of genin in 10 parts C<sub>6</sub>H<sub>5</sub>N was treated with 2 c.c. BzCl; much heat was evolved and a very strong red-violet colour developed; 40 parts H<sub>2</sub>O added after 20 hours gave a dirty red precipitate, becoming brick-red after washing and drying *in vacuo*; the product could not be purified. A pure *dibenzoate*, fine needles, sinters about 190°, is obtained, however, when 0.78 Gm. of genin in 50 parts cold C<sub>6</sub>H<sub>5</sub>N is slowly treated with 2 c.c. BzCl, then, after 18 hours, with 75 parts H<sub>2</sub>O, filtered from the tarry precipitate, treated with 25 parts more of H<sub>2</sub>O and allowed to stand overnight. The sugar solution obtained in the preparation of genin is freed of HCl with Ag<sub>2</sub>CO<sub>3</sub>, concentrated at 35°C. to remove the EtOH, moderately diluted with H<sub>2</sub>O, shaken twice with CHCl<sub>3</sub> to remove the last traces of tarry impurities and decomposition products and concentrated to a syrup *in vacuo* over H<sub>2</sub>SO<sub>4</sub>; on sowing with digitoxose, about 0.5 crystallizes; about 0.5 as much again can be obtained from the

mother liquors by dissolving them in a little absolute EtOH, adding 2 volumes of Et<sub>2</sub>O (which gives a precipitate rich in ash) and again evaporating to a syrup and keeping over KOH; 70 Gm. of digitoxose were thus obtained. The final mother liquors (about 65 Gm.) consist essentially of digitoxose but cannot be made to crystallize, affording a remarkable instance of the influence of small amounts of impurities in preventing crystallization. The oxidation of digitoxose to dihydroxy-glutaric acid digitoxose can be carried out directly, without first preparing the digitoxonic acid, by heating digitoxose with 5 parts dilute HNO<sub>3</sub> (sp.g. 1.2) for 12 hours at 30-2°, then 24 hours at 50°, then quickly to 70° and 10 hours at 70-90°; the traces of distinctly crystalline and difficultly soluble Ca salt observed as byproduct in the earlier work have now been identified as Ca mesotartrate digitoxose; therefore, the corresponding HO groups in dihydroxy-glutaric acid, and in digitoxose itself must occupy the meso position. From the amount of genin obtained on cleavage, the author believes that the glucoside corresponding to it contains two digitoxose residues and, therefore, has the composition C<sub>34</sub>H<sub>52</sub>O<sub>12</sub>.

**Anthraquinones, The Microchemistry of several.** E. Senft. (*Z. öster. Apoth.-Ver.*, 52, 165-6, 181, 201; *Chem. Abstr.*, 1914, 8, 3616.) For the microchemical examination of anthraquinone derivatives, lichens offer exceptionally suitable material. The solubility in various solvents, the form of the crystals, their optical and other physical properties, such as m.p., may all be considered. The author reports on rodocladonic acid found in various species of *Cladonia*, solorinic acid found in *Solorina crocea*, rhodophysein, obtained from *Physica endococcinea*, and blastenin found by Hesse in *Blastenia arenaria* and *Blastenia percrocata*.

**Beer Yeast for Industrial and Therapeutic Purposes.** E. Carlinfant. (*Annali Chim. appl.*, 1914, 2, 121; *J.S.C.I.*, 1914, 33, 1068.) In determining the activity of yeast by a fermentation test, considerably higher results are obtained if the CO<sub>2</sub> be removed continuously by a current of air during the test. Freshly prepared dried yeast shows a considerably lower fermenting power than fresh yeast in the first 12 hours of the test, but its activity subsequently increases. When dried yeast is kept, its fermenting power diminishes and may almost completely disappear. A number of commercial

preparations of dried yeast for pharmaceutical use were examined. In all cases scarcely any fermentation occurred in the first 12 hours, and the fermenting power was very low even after 24 hours.

**Beta-mono-dextro-galactoside of Ethylene Glycol, Biochemical Synthesis of.** E. Bourquelot, M. Bridel and A. Aubry. (*Comptes rend.*, 1915, 160, 571.) By prolonged contact, at first for some 3 months at 33°C. and then for several months at the laboratory temperature, of emulsin with an aqueous solution of ethylene glycol and galactose, the  $\beta$ -mono-galactoside of ethylene glycol has been obtained. When optical examination showed that synthesis had stopped, the excess of galactose was removed by fermentation with yeast and added glucose: uncombined glycol was eliminated by shaking out with acetic ether. The  $\beta$ -galactoside was then precipitated from EtOH solution by means of Et<sub>2</sub>O. The precipitate thus formed quickly assumed a definite crystalline structure, and needles were deposited from the EtOH-Et<sub>2</sub>O mother liquor. These had a sweetish taste; m.p. 133°–134°C.; were optically inactive, and failed to reduce Fehling's solution. When hydrolyzed with dilute H<sub>2</sub>SO<sub>4</sub> or with emulsin, they yielded only one molecule of galactose, thus proving that the compound was the  $\beta$ -mono-galactoside.

**Beta-monoglucoside of Ordinary Propylene Glycol, Biochemical Synthesis of.** A. Aubry. (*Comptes rend.*, 1915, 160, 214.) On warming together isopropyleneglycol, glucose and emulsin, in presence of a little water, then abandoning the mixture to the ordinary laboratory temperature for 6 months, synthesis is effected by the enzyme,  $\beta$ -propylene glycol monoglucoside being formed. This is soluble in alcohol, from which solution it is precipitated on adding ether. In this manner the glucoside may be obtained pure, in the form of a nearly solid, amorphous, white mass; its optical rotation is  $-30^{\circ}32'$ . It is hydrolyzed by emulsin in presence of water, and the liberated glycol, like the original substance, is optically inactive. Since isopropylene glycol is known to be a racemic compound, consisting of lævo- and dextro-glycols of equal optical activity in either direction, the enzyme must exert its synthetizing power equally on both the lævo- and dextro-constituents



since the isopropylene glycol liberated from the glucoside is also devoid of rotatory power.

**Canvalia ensiformis Seeds, Urease in.** H. E. Annet. (*Biochem. J.*, 1914, 8, 449; *Chem. Abstr.*, 1915, 9, 641.) The seeds of the Indian sword-bean, *Canvalia ensiformis*, contain much more urease than any of six specimens of soya-beans examined. Ten Gm. of powdered seed was treated with 100 c.c. distilled water and allowed to stand with occasional shaking for 1 hour at room temperature in presence of toluene. Two c.c. of this extract was added to 50 c.c. of a 1 per cent. urea solution together with 0.5 c.c. toluene. Five c.c. of the liquid was now immediately removed and titrated with N/10 HCl, using methyl orange as indicator. Then, generally at half-hour intervals, successive 5 c.c. portions were titrated. The tests were carried on at about 27°C.

**Castor Oil Seeds, Urease of.** K. G. Falk and K. Sugiyra. (*J. Am. Chem. Soc.*, 1914, 36, 2166.) Castor oil seed preparations hydrolyzed much less urea than similar soya-bean preparations under comparable conditions. It is, therefore, considered that either castor oil seed urease is less active than the soya-bean enzyme, or that less urease is present in castor oil seeds than in soya-beans. The influence of acids, bases, and salts on the hydrolytic action of the urease of the two seeds is similar.

**Enzymes, Behaviour of, at Low Temperatures.** J. S. Heburn. (*J. Amer. Pharm. Assoc.*, 1915, 4, 682.) A review of the published and original observations on the subject is thus summarized: The power to survive prolonged exposure to low temperatures is possessed by various enzymes, including those producing hydrolysis of fats, of carbohydrates, and of proteins, those concerned in biochemical oxidations and reductions, the clotting enzymes and that of alcoholic fermentation. The enzymes retained their catalytic power after exposure, either *in situ* or in solution *in vitro*, to temperatures varying from a few degrees above 0°C. to the temperature of liquid air (−180° to −191°C.). The shortest periods of holding, invariably less than one day and usually less than one hour, were at the temperature of liquid air. The longest period of holding was 89 months at a temperature of −9.4° to −12.2°C. The activity of certain of these enzymes, including rennin, zymase, and those hydrolyzing fats, carbohydrates, and proteins, has been studied

at low temperatures, varying from that of an ice-box to one of  $-9^{\circ}$  to  $-12^{\circ}\text{C}$ . While the enzymes produced autolytic digestion or acted on artificial media at these temperatures, the velocity of the reaction was always lessened to a considerable degree.

**Ethylene Glycol  $\alpha$ -mono-dextro-galactoside, Biochemical Synthesis of.** E. B o u r q u e l o t, M. B r i d e l and A. A u b r y. (*Comptes rend.*, 1915, 160, 674.) By leaving in contact for 9 months ethylene glycol, galactose, dried bottom yeast and an appropriate amount of water,  $\alpha$ -mono-dextro-galactoside,  $\text{C}_6\text{H}_{11}\text{O}_5\cdot\text{OCH}_2\cdot\text{CH}_2\text{OH}$ , has been obtained in colourless rosettes of faintly sweetish needles; m.p.  $134^{\circ}\text{C}$ . (like its  $\beta$ -stereoisomer);  $\alpha_D + 169.9^{\circ}$ ; soluble in water and in EtOH; hydrolyzed by dilute  $\text{H}_2\text{SO}_4$ , and, very slowly, by  $\alpha$ -glucosidase of bottom yeast. To isolate it, the reaction mixture was treated first with EtOH and  $\text{Et}_2\text{O}$  which precipitated the uncombined ingredients and some by-products. The liquid, freed from these precipitants, was evaporated and the residue extracted with acetone. After removing this solvent, the residue was distilled *in vacuo* to get rid of the uncombined glycol. The black syrupy distillation residue soon became crystalline. In spite of unavoidable decomposition products, the galactoside was ultimately extracted from this by means of absolute EtOH and purified with animal charcoal and by recrystallization from EtOH.

**Ferment Reactions, Influence of Silent Electric Discharge on.** W. L o e b and A. S a t o. (*Biochem. Zeit.*, 1915, 9, 1490; *Chem. Abstr.*, 1915, 9, 1490.) Aqueous solutions of starch are hydrolyzed by the action of the silent discharge both in the presence and absence of O. The part of the starch not hydrolyzed in this process is changed, possibly polymerized, so that it is more stable towards diastase than the untreated starch. The electrical treatment of pancreatin solutions retards considerably their diastasic properties. In the same way the reaction between diastase and starch is retarded. Milk peptone solutions are hydrolyzed only to a very slight degree, giving free  $\text{NH}_3$ . Casein and fibrin are stable. The tryptic properties of pancreatin solutions are retarded by the discharge. The reaction between enzymes and casein is slightly retarded. Tributyrin is hydrolyzed by the action of the silent discharge. The lipase of the pancreatin is weakened by it.

**Gentiobiose.** G. Z e m p l é n. (*Ber.*, 1915, 48, 233; *J.S.C.I.*, 1915, 34, 567.) The biochemical synthesis of gentiobiose by the action of emulsin on dextrose, which was achieved by Bourquelot, Hérissé, and Coirre (*Y.B.*, 1914, 92), does not accord with E. F. Armstrong's statement that maltose may be obtained in this way. The formation of an  $\alpha$ -disaccharide, like maltose, is also contrary to the established rule that enzymes only bring about the synthesis of those disaccharides which, under other conditions, are hydrolyzed in their presence. In the case of emulsin these would be  $\beta$ -disaccharides like gentiobiose, cellobiose, or *iso*-maltose. The author repeated Bourquelot's work with positive results, for he isolated the octa-acetate and phenyl osazone of gentiobiose from the product of the action of emulsin on a 50 per cent. dextrose solution. With the idea of deciding whether gentiobiose is identical with *iso*-maltose or not, syrups containing the latter were prepared by Fischer's method and acetylated. Although the octa-acetate of gentiobiose is readily isolated from very impure products, it could not be obtained from these syrups, and it seems to be highly probable, therefore, that gentiobiose and *iso*-maltose are not identical.

**Glucose, Commercial, and its Uses.** G. W. R o l f e. (*Amer. J. Pharm.*, 1915, 87, 269.) A strong plea for the wider use of syrupy glucose as a valuable and pure food carbohydrate. Contrary to the general opinion, commercial glucose is not mainly composed of dextrose. As a rule, it contains 20 per cent. of dextrose, 45 per cent. of maltose, and 35 per cent. of dextrans. The use of glucose in syrups, jams and confectionery should be encouraged since, especially in conjunction with cane sugar, it has many advantages and is a cheap and valuable food. A description of the process for preparing glucose by the hydrolysis of maize starch with HCl is given.

**Glucosides, Tannins, and their Decomposition Products, Action of, on Germinating Seeds.** W. S i g m u n d. (*Biochem. Zeit.*, 62, 339; *Chem. Abstr.*, 1914, 8, 2561.) Arbutin is little injurious for germination, 0.7 mol. per litre of  $H_2O$  having only a slight effect. Hydroquinone is much more poisonous, 0.1 mol. being a lethal dose, while the sugar component, *d*-glucose, has no effect in a concentration of 0.8 mol. per litre. Phloridzin, hesperidin, phloretin and hesperetin have little effect. Baptisin is a strong poison, acting in a concentration of 0.002 mol.

per litre. Salicin is slightly injurious in concentrations of 0.02 mol., while helicin is more harmful. Populin, as far as it is soluble in cold  $H_2O$ , has no effect. Coniferin is little poisonous; decomposed by emulsin it becomes more toxic. Syringin and amygdalin have little effect. Esculin is poisonous in proportions of 0.004 mol. per litre. Sinigrin and convallarin are not harmful. Helleborein, 0.2 and 0.4 per cent., is rather poisonous, also digitalin, saponin and sapogenin. Strophanthin is poisonous. Phenol is poisonous in concentration of 0.02 mols.; pyrocatechol, resorcinol and pyrogallol are very poisonous. Quinone in a concentration of 0.02 mol. per litre is fatal. Saligenin, salicylaldehyde, benzaldehyde, vanillin, piperonal; benzoic, salicylic, cinnamic, *o*-cumaric acids; esculetin, daphnetin; mandelic, protocatechuic, gallic acids; tannin, catechin, catechutannic acid were also tested and the figures are given.

**Glucosides in Leguminosae and Scrophulariaceae.** E. Bourquelot and Adèle Fichtenholz. (*J. Pharm. Chim.*, 1915, 11, 219.) The presence of one or more glucosides is indicated by the biological method in the following plants —*Cytisus laburnum*, *Crionis natrix*, *Psoralea bituminosa*, *Indigofera leptostachys*, *Scrophularia aquatica*, *Linaria spuria*, *L. elatine*, *L. cymbalaria*, *L. vulgaris*, *L. purpurea*, and *Euphrasia officinalis* (see also *Y.B.*, 1911, 121). In all these, except *Linaria spuria*, cane sugar is present as well. In this plant the optical deviation indicates that the accompanying sugar is not sucrose. In *Coronilla vera* and in *Melampyrum arvense* no evidence of the presence of glucoside was obtained. The *Linariae* are particularly rich in glucosidal material. This confirms the work of Klobb (*Y.B.*, 1907, 97; 1908, 105), who has found two glucosides, one of which is insoluble in water, in *Linaria vulgaris*. (See also *Y.B.*, 1914, 90.)

**Glucosides, Formation of, from Glycerin by means of Emulsin.** E. Bourquelot, M. Bridel and A. Aubry. (*Comptes rend.*, 1915, 160, 823.) At least two  $\beta$ -monoglucosides are formed by the prolonged contact of glucose, glycerol, emulsin (otherwise  $\beta$ -glucosidase) and a little water. After 10 months' contact the mixture, which was originally strongly dextrorotatory, had gradually become slightly laevogyre. When no further optical change was apparent, the uncombined glucose was removed by fermentation with yeast. Free glycerol was eliminated by means

of a number of successive washings with a mixture of pure acetone containing 10 per cent. by volume of EtOH, 95 per cent. Glycerol is soluble in this mixture, but the glucosides are insoluble. The thick syrupy mass was then further purified by solution in EtOH and precipitation with acetic ether. Although the glucosides comprising the reaction product have not yet been isolated in a crystalline condition, the evidence afforded by the optical rotation of the solutions, after hydrolysis either with dilute  $\text{H}_2\text{SO}_4$  or with emulsin, and the amount of glucose so liberated, shows conclusively that at least two  $\beta$ -monoglycosides of glycerol have been formed.

**Glycerin, Action of, on Alcoholic Fermentation and on Invertin.**

G. Rossi. (*Boll. Chim. Farm.*, 1914, **53**, 687; *J. Pharm. Chim.*, 1915, **11**, 243.) Glycerin will prevent alcoholic fermentation only when it is present in relatively large quantities. To totally arrest the action of yeasts not less than 42 per cent. of glycerin must be present in the liquid. When the amount of glycerin falls below 10.6 per cent., it is without any influence even on the rate of fermentation. Although yeast is killed by 50 per cent. of glycerin, its invertin still retains its specific action.

**Helianthus annuus, Presence of Glucoside in.** A. Zanotti.

(*Boll. Chim. Farm.*, 1914, **53**, 4; *Chem. Abstr.*, 1915, **9**, 1483.) The aqueous extract of sunflower stems, purified by treatment with basic lead acetate, then with EtOH, gave evidence of presence of a glucoside, for which the formula  $\text{C}_{11}\text{H}_{19}\text{O}_4\text{N}_2$  is suggested.

**Helleborein.** E. Sieburg. (*Archiv. Pharm.*, 1914, **151**, 154.) The helleborein examined was a colloidal powder, readily soluble in water giving a neutral solution  $[\alpha]_{\text{d}_{22}} - 2.8^\circ$ . A number of colour reactions and precipitants are described. Helleborein is a saponin having the composition  $(\text{C}_{21}\text{H}_{34}\text{O}_{10})_3$ . Its acetyl and benzoyl derivatives are described. Dilute acids hydrolyze helleborein; acetic acid, glucose, a neutral and an acid helleboretin being formed, the solution acquiring a deep violet colour. Taka-diastase and ricin lipase effect a similar hydrolysis. The acid and neutral helleboretins thus formed are separated by means of acetic ether. The characters and colour reactions of both are fully described. From a consideration of these helleborein is classed among the chromogenic saponins. Deacetylated

helleborein, obtained by the saponification of acetyl-helleborein, is non-toxic. Although pharmacologically helleborein cannot be considered to be a substitute for digitalis, it undoubtedly forms a connecting link between the saponins and the digitalis glucosides. (See also *Y.B.*, 1913, 133.)

**Kombé-strophanthin, Crystalline.** D. H. Brauns and O. E. Closson. (*Arch. Pharm.*, 1914, 252, 294; *Chem. Abstr.*, 1915, 9, 507.) The seeds of *Strophanthus kombé* contain 2 strophanthins; a crystalline glucoside (a),  $C_{40}H_{56}O_{15} \cdot 3H_2O$ , and a closely related amorphous strophanthin (b), which possesses apparently double the mol. wt. of (a). (a) is converted by  $H_2O$  into a monobasic acid amorphous strophanthin (c). It is likewise possible that such action is productive of a mixture of (c), of a dibasic acid strophanthin and of (a). These 3 strophanthins yield with dilute acids strophanthidin,  $C_{27}H_{38}O_7 \cdot H_2O$ , identical with that described by Feist and also by Heffter and Sachs. (a) contains neither a pentose nor a methyl-pentose (rhamnose). (b) appears to contain a pentose. The crystalline Kombé-strophanthin which Arnaud prepared is unquestionably identical with that obtained by the authors. It was further established that the hydrate used by Arnaud in the elementary analysis is a new chemical derivative of the original strophanthin. In the present paper it is designated (c). The results obtained by Kohn and Kulisch are in large measure corroborative of those described by the authors for (c). The data relative to the formers' strophanthidin however differ from those given by the authors. The difference may possibly be explained in another kind of hydrolysis of strophanthin or of a change in crystallizing the strophanthidin. The strophanthin examined by Fraser and likewise by Feist is very probably identical with the amorphous preparations prepared by Heffter and Sachs, as also with those obtained by the authors according to Gerrard's method. (a) is apparently split by dilute acids as indicated in the equation:  $C_{40}H_{56}O_{15} + 4H_2O = C_{27}H_{38}O_7$  (strophanthidin) +  $C_{12}H_{22}O_{11}$  (disaccharide) +  $CH_3OH$ . Although the purity of an amorphous substance is more or less open to question, it was nevertheless found that an amorphous strophanthin, in addition to (a), must be present in identified *S. kombé* seeds. Heffter and Sachs have shown that identified *S. hispidus* seeds contain no crystalline strophanthin, but an amorphous variety identical with or closely related to (b). Both (a) and (c) show the typical action of a

heart tonic, lessening the count but increasing the amplitude of pulse together with a slight increase in blood pressure. The action of (c) is less than that of the crystalline variety. The activity of these substances determined according to Houghten's method was found to be as 1 : 3. The fact that this loss in physiological activity is associated with the loss of a lactone group appears to be sufficiently interesting to note. Since (a) represents one of the active constituents of *Strophanthus kombé* seeds and, as a crystalline substance, possesses a definite activity, the suggestion is made that this compound should serve as a standard for measuring the activity of various preparations of the drug. (See also *Y.B.*, 1913, 134.)

**Lactose in Milk, Determination of, Colloidal Iron for the Precipitation of the Proteins in.** R. L. Hill. (*J. Amer. Pharm. Assoc.*, 1915, 4, 744.) To a 10 Gm. sample of milk, which has been diluted to about 25 c.c., about 3 c.c. of a 10 per cent. solution of colloidal iron (dialyzed ferric hydroxide) are added. The amount of colloidal iron necessary depends upon the composition of the milk and can be accurately determined by adding the last portion drop by drop, and agitating after each addition. If the precipitation is complete, a clear supernatant liquid separates out from the flocculent precipitate; if too little has been added, the supernatant liquid will appear milky; if too much, it will have a reddish tinge. The sample is next filtered into a 100 c.c. volumetric flask, and the precipitate thoroughly washed with distilled water until the filtrate and washings aggregate about 100 c.c. The flask is then filled to the mark and the percentage of lactose determined by Benedict's quantitative method. About 16 c.c. of the diluted sample will be required to reduce completely 25 c.c. of Benedict's quantitative solution. A very convenient method of analysis is given by Cole, in which a 4 ounce flask is used instead of an evaporating dish. The wide-mouthed Jena 150 c.c. flat-bottomed flasks are very convenient for the determination. Three to 4 Gm. of anhydrous  $\text{Na}_2\text{CO}_3$  is dissolved, by means of heat, in 25 c.c. of twice diluted Benedict's solution, to which a little powdered pumice has been added. About 14 c.c. of the sugar solution is then rapidly added from a burette. Boiling is continued for at least half a minute before the addition of more lactose solution. When reduction is complete the supernatant liquid will have a slight yellowish tinge to which the blue colour very slowly returns. If the end-point has been under-

estimated, it will have a blue or greenish tinge that rapidly becomes bluer. With a little practice, and by adding the last portion a drop at a time, and boiling half a minute after each addition, the end-point can be determined to within one drop. Twenty-five c.c. of Benedict's quantitative solution are completely reduced by 0.0676 of a Gm. of anhydrous lactose. Since the milk has been ten-fold diluted, 0.0676 divided by the number of c.c. of diluted lactose solution used, multiplied by ten, will give the percentage of lactose in the milk.

**Lophopetalum toxicum, Crystalline Glucoside from.** Galvialo. (*Petrograd P.J.*, 1914, 515; *J. Pharm. Chim.*, 1915, 11, 79.) The bark of *Lophopetalum toxicum* is used by the Philippinos as an arrow poison. The powdered bark was first extracted with petroleum ether to remove waxy and other impurities. It was then extracted by boiling in EtOH 90 per cent. with a little KOH. The residue obtained on distilling off the EtOH was then treated with Et<sub>2</sub>O which gave a yellow crystalline residue. This was finally purified by boiling EtOH 95 per cent. and animal charcoal and subsequent recrystallization. In this manner the glucoside *lophopetalin*, C<sub>36</sub>H<sub>54</sub>O<sub>8</sub>, was obtained in long, colourless needles; insoluble in water; readily soluble in hot, less so in cold EtOH; soluble in Et<sub>2</sub>O, CHCl<sub>3</sub> and benzin. The m.p. varies with the solvent from which it has been crystallized; from benzin, 222° to 228°C.; from EtOH 95 per cent., 225° to 230°C.; from Et<sub>2</sub>O, 190° to 195°C. The yield is 0.2 per cent. Its physiological action has not yet been determined.

**Mycogalactan, a New Polysaccharide in Aspergillus niger.** A. W. Dox and R. E. Neidig. (*J. Biol. Chem.*, 19, 235-7; *Chem. Abstr.*, 1914, 8, 3668.) Mycogalactan, obtained from cultures of *A. niger*, is a white powder, swelling then dissolving in water. The solution gives a faint blue colour with I. For a 0.5 per cent. solution  $[\alpha]_D^{20} = +284^\circ$ . After hydrolysis with HCl the solution was laevorotatory and reduced Fehling solution, yielding galactose.

**Olea europea Fruit, Bitter Principles of.** F. T. Bioletti. (*California Sta. Rept.*, 1914, 197; *Chem. Abstr.*, 1915, 9, 1350.) The bitter principles are soluble in water, hot EtOH, and CHCl<sub>3</sub> and slightly in Et<sub>2</sub>O. The bitterness is not done away with by exact neutralization, by the presence of a slight excess of alkali, by exact neutralization and heating under 15 lb. pressure for



one hour, by a slight excess of HCl and heating under pressure, by heating untreated juice under pressure, by the presence of an excess of  $\text{NaHCO}_3$ , or by fermentation with yeast. It is destroyed by using a considerable excess of NaOH or  $\text{Na}_2\text{CO}_3$  or a slight excess of alkali and heating under pressure. The tests on juice alone showed that a 2 per cent. KOH solution will destroy the bitterness immediately, and neutralization and the addition of 0.7 per cent. excess of KOH, within 24 hours. An excess of 0.56 per cent. of alkali had little effect. The glucoside, oleuropein, appears to be the cause of the bitterness of olives. (See also *Y.B.*, 1908, 148; 1909, 64; 1910, 113.)

**Oxymethylantraquinones, Qualitative Separation and Identification of.** E. M. Bailey. (*Amer. J. Pharm.*, 1915, 87, 145.) By taking advantage of differences of solubility in alkaline solutions of certain oxymethylantraquinones and the colour reactions of these when liberated by acids, the author has been able to differentiate them, and to a certain extent the cathartic drugs from which they may be derived. Twenty-five c.c. of fluid extract of the various drugs examined were evaporated to remove EtOH, diluted with 23 c.c. of water and treated with an excess of  $\text{Pb } 2(\text{C}_2\text{H}_3\text{O}_2)$  and filtered. The lead precipitate was digested for one hour with 10 per cent.  $\text{H}_2\text{SO}_4$  in a boiling water-bath. The solution was filtered and the filtrate extracted, while still hot, with hot  $\text{C}_6\text{H}_6$ . In the case of powdered drugs, 3 Gm. of material was boiled with alcoholic KOH under a reflux condenser for one hour. The solution was then evaporated to remove EtOH, diluted with 50 c.c. of water, acidified with dilute  $\text{H}_2\text{SO}_4$ , and extracted directly with hot  $\text{C}_6\text{H}_6$ .

By washing the benzol solution first with 5 per cent.  $\text{Na}_2\text{CO}_3$  solution and then with 5 per cent. NaOH, emodin and chrysophanic acid can be separated. The separation can be made quite sharp if one bears in mind that emodin is very readily soluble in  $\text{Na}_2\text{CO}_3$ , and that chrysophanic acid is slightly soluble also. Two or three washings of the  $\text{C}_6\text{H}_6$  solution with  $\text{Na}_2\text{CO}_3$  are usually sufficient to remove emodin, and the aqueous solution is of deep red colour. As the  $\text{C}_6\text{H}_6$  is further washed with this reagent, the washings become pink, due to the slight solubility of chrysophanic acid. Treatment with  $\text{Na}_2\text{CO}_3$ , then, should be discontinued when the washings become pink. The chrysophanic acid can be readily removed by one or two washings with NaOH. In a few

preliminary experiments it was found that an initial treatment of the benzol solution with 5 per cent.  $\text{Am}_2\text{CO}_3$  removed a considerable amount of colouring matter which did not subsequently behave like either emodin or chrysophanic acid, but did, in most cases, give the test for oxymethylantraquinones. On account of this fact, and since chrysophanic acid is practically insoluble in dilute  $\text{Am}_2\text{CO}_3$ , and emodin but slightly soluble, the addition of  $\text{Am}_2\text{CO}_3$  to the series of reagents for the fractional washing of the  $\text{C}_6\text{H}_6$  extract suggested itself.

This  $\text{C}_6\text{H}_6$  extract was shaken out in a separator with 25 c.c. portions of  $\text{Am}_2\text{CO}_3$  1 : 20 solution until colourless or faintly coloured; then with similar portions of  $\text{Na}_2\text{CO}_3$  1 : 20 solution; and finally with  $\text{NaOH}$  1 : 20 solution. The three separate alkaline aqueous extracts were then acidified; shaken out with  $\text{Et}_2\text{O}$ , the  $\text{Et}_2\text{O}$  solution separated, the solvent evaporated and the residues tested with  $\text{H}_2\text{SO}_4$ ,  $\text{HNO}_3$  and water added in the order indicated. The colour reactions obtained from the  $\text{Et}_2\text{O}$  residue of the methylantraquinones liberated by acid from the  $\text{Na}_2\text{CO}_3$  washings may be summarized as follows:—

Emodin, as derived from buckthorn, rhubarb, and senna: with concentrated  $\text{H}_2\text{SO}_4$ =pink; +  $\text{HNO}_3$ =yellow; +  $\text{H}_2\text{O}$ =pink solution. As derived from aloes (sodium carbonate soluble): with concentrated  $\text{H}_2\text{SO}_4$ =red, brownish; +  $\text{HNO}_3$ =yellow; +  $\text{H}_2\text{O}$ =yellow solution. Chrysophanic acid, as derived from all sources: with concentrated  $\text{H}_2\text{SO}_4$ =orange red; +  $\text{HNO}_3$ =yellow; +  $\text{H}_2\text{O}$ =yellow solution and precipitate. Unidentified oxymethylantraquinones (ammonium carbonate soluble): with concentrated  $\text{H}_2\text{SO}_4$ =purple or violet; +  $\text{HNO}_3$ =yellow; +  $\text{H}_2\text{O}$ =yellow solution.

A relatively large amount of chrysophanic acid was present in the aloes examined. The colour removed by  $\text{NaOH}$  was greatly in excess of that removed by  $\text{Na}_2\text{CO}_3$ .

The tests were applied thus to the  $\text{Et}_2\text{O}$  residue evaporated in a white porcelain capsule: 4 to 5 drops of strong  $\text{H}_2\text{SO}_4$ , then 1 or 2 drops of  $\text{HNO}_3$ ; and finally about 1 c.c. of water. The nature of the substances separated from aloes, under these conditions, by  $\text{Na}_2\text{CO}_3$  and from all the drugs investigated by  $\text{Am}_2\text{CO}_3$ , is under investigation.

**Papain, Assay of.**—Th o r b u r n. (*J. Amer. Pharm. Assoc.*, 1915, 4, 223.) Dissolve papain 0.400 Gm. and  $\text{NaHCO}_3$  0.750 Gm.

in distilled water enough for 100 c.c. Heat to 50°–55°C. Scrape lean rump steak (better results are obtained by scraping the meat to a pulp instead of grinding), rejecting gristle, fat, etc., to a pulp; weigh 10 Gm. meat pulp and place in a 200 c.c. digestion-flask; add 100 c.c. of the warm solution of papain and  $\text{NaHCO}_3$ . Digest for 4 hours, shaking the mixture once every 10 minutes; then pour into a measuring cylinder and let stand at rest for half an hour. If the digestion flask is fitted with a stopper carrying a small graduated tube it is of course much more convenient than pouring into a cylinder; in this case invert the flask and read after half an hour's standing. A blank digestion of the meat pulp with  $\text{NaHCO}_3$  should be carried along with the papain digestion. Not more than 10 c.c. of residue should remain after the alkaline digestion with papain. After reading this residue, warm the mixture to 50°–55°C. and add concentrated  $\text{HCl}$  1.5 c.c.—sufficient to neutralize the alkali and leave 0.2 per cent. to 0.3 per cent. of free acid. Again digest for 4 hours, shaking every 10 minutes. Let stand at rest half an hour, then read the residue which should be less than 3 c.c.; this gives the total digestive power of the papain as 1 to 25 four-hour tests which compares favourably with a 1 to 30 six-hour test.

**Papain, Commercial, Standardization of.** F. W. Heyl, C. R. Caryl and J. F. Staley. (*Amer. J. Pharm.*, 1914, 86, 543.) The reagents required are: (a) Egg albumin solution. The separated whites of six fresh eggs are slightly beaten, and diluted with 2 volumes of  $\text{NaCl}$  solution 1 : 100. Mix and filter through paper. Make up to a definite volume so that 15 c.c. contains 0.400 Gm. of coagulable protein as found by experiment on the strong filtrate. (b) Weigh 1 Gm. of the papain and transfer into a dry 100 c.c. graduated flask. When everything else is ready for the determination, the papain is taken up with some 1 : 100  $\text{NaCl}$  solution, shaken thoroughly, and made up to 100 c.c. Exactly 30 minutes should elapse from the time the  $\text{NaCl}$  solution is poured on the papain until the aliquot parts of the solution are taken. (c)  $\text{N}/2$  acetic acid. *Determination of proteolytic activity at 80°–100°C.*—Into a dry 50 c.c. Erlenmeyer flask place 15 c.c. of the standard albumin solution, 1 c.c. of the papain solution and 9 c.c. of  $\text{NaCl}$  solution. Transfer at once to the thermostat, already regulated at 80°C., and allow the digestion to proceed for exactly 15 minutes. Now add 1 c.c.

of N/2 acetic acid and transfer immediately to a bath at 100°C. and heat for 10 minutes. The time factor should be given the sharpest attention. Bath at 80°C., 15 minutes. Transfer, 1 minute. Bath at 100°C., 10 minutes. In order to facilitate the acidification, a two-holed stopper is used, bearing a long glass tube to serve as a condenser, and a small funnel into which 1 c.c. of acetic acid can be easily placed. The undigested protein is filtered off on a tared filter paper. Wash free from chlorides. Wash with 10 c.c. of 95 per cent. alcohol, and when this has passed through add 10 c.c. of ether U.S.P. Dry at 100°–105°C. to constant weight.

At the same time that the above digestion is carried out, the amount of protein in the egg-white solution coagulable by heat is determined in a blank, i.e., 15 c.c. of the same egg-white solution is mixed with 10 c.c. salt solution (or, better, 9 c.c. salt solution and 1 c.c. of the papain solution in which the enzyme has been destroyed by boiling vigorously for 15 minutes), and the operations are carried out upon this mixture exactly as described above. Calculate the percentage of protein rendered non-coagulable under these conditions.

*Test for Pepsin in Papain.*—Take 15 c.c. of the same egg-white solution as prepared for the first digestion. Add 2 c.c. 1 per cent. salt solution, 3 c.c. of N/2 HCl and, lastly, 5 c.c. of a 1 per cent. papain solution. Add 0.5 c.c. toluol to prevent putrefaction. Digest at 40°C. for 15 hours. Add 25 c.c. of a 10 per cent. solution of trichloroacetic acid. Heat to boiling on an electric stove. Boil ten minutes and filter through a tared paper, and wash the coagulum free from acid. Wash with alcohol and ether. Dry at 100°–105°C. to constant weight. At the same time that this digestion is carried out the total amount of coagulable protein present should be determined in a blank experiment.

A series of experiments with dried papaw juice are described, and numerous tables of results are given. In these it is shown that the digestion proceeds rapidly at 80°–100°C. This characteristic property can be utilized for the standardization of commercial papain samples. Under the conditions outlined above, dried papaw juice should be capable of dissolving at 80°–100°C. not less than 40 per cent. of the egg-albumin taken. No samples of "papain" were found upon the market which had a higher digestive activity than the samples of dried papaw latex under the conditions employed. Since the use of the term "papain" is misleading, papain products ought to be

marketed as "dried papaw juice," and that only a lower limit of digestive strength should be stated in defining a standard for it. A definition proposed upon this basis might be stated as follows: Dried papaw juice is the dried albuminous exudate of the fruit of *Carica papaya*, free from starch, sugars, and diluents, and contains a proteolytic enzyme or enzymes. When assayed by the method above it has the power of digesting at 80°–100°C. not less than 40 per cent. of the unaltered egg-white protein. Of 26 samples studied, 7 represented the undiluted dried latex, 15 contained starch in amounts varying from 15 per cent. to 58 per cent., while 3 were diluted with sugar and one with dextrin. Four samples showed a high digestive strength under conditions favourable for pepsin digestion. On the basis of the standard proposed above, 12 samples, or 44 per cent., have been diluted to such an extent that their digestive strength is below a very reasonable requirement. (See also *Gen. Index* and *Y.B.*, 1911, 181; 1913, 141; 1914, 98.)

**Papain, Its Production and Commerce.** H. F. Mac Millan. *Chem. & Drugg.*, 1915, 86, 133.) An interesting illustrated article dealing with the history of the Papaw tree, *Carica papaya*, and the smaller mountain papaw, *C. candamarcensis*, grown in Ceylon, where the latter, originally introduced from Ecuador, is now semi-naturalized. Papain is collected from *C. papaya* only in Ceylon. Many details of interest are given in the communication. (See also *Y.B.*, 1914, 98.)

**Sugar, the Nipa Palm as a Commercial Source of.** D. Pratt, L. N. Thurlow, R. R. Williams and H. D. Gibbs. *Philipp. J. Sci.*, (A) 8, 377; *Chem. Abstr.*, 1914, 8, 3728.) The nipa palm is better than other palms as it grows in waste land, and the stalk from which the juice comes is close to the ground. The palm produces about 40 litres of sap per tree during a season of 6 months. The maximum flow is during the second month. The juice averages 15 per cent. sucrose, with an apparent purity of 85 and contains only traces of reducing sugars, with no waxes, acids, pectins, etc. The enzymes of the invertase and peroxidase types, are extremely active. To collect the sap and preserve it, bamboo joints are used after washing out with lime cream and SO<sub>2</sub>. The latter preserves the sap. 115 kg. of 99–99.5° sugar can be made from 1,000 litres of sap

without any important modification in the usual method. One hectare of nipa swamp, yielding 30,000 litres of sap per season, should produce 200–250 litres per day, therefore it would require about 450 hectares of good swamp to supply a 10 ton mill with its daily requirements of raw material.

**Saponin of *Styrax japonica*.** Y. Asahina and M. Momoya. (*Arch. Pharm.*, 252, 56; *Chem. Abstr.*) The crystals previously obtained by Keimatsu were found to be not the saponin itself but its Ca salt. The pure saponin (jegosaponin), was prepared by boiling the fruit shells with 95 per cent. MeOH, evaporating filtrate until residue began to foam, shaking the finely powdered crystals (which separated during the course of 24 hours and contain 3 per cent. of ash) with cold 0.5 per cent. HCl and recrystallizing the product from MeOH. *Jegosaponin*,  $C_{55}H_{80}O_{25}$ , forms colourless needles m.p. 238°C. insoluble in water,  $Et_2O$ ,  $C_6H_6$  and  $CHCl_3$ , quite soluble in warm EtOH, MeOH and cold AcOH.  $[\alpha]_D = -39.15^\circ$ . On shaking with water, the pure saponin does not foam, it acquires this property however on the addition of a little alkali or alcohol. With  $H_2SO_4$  a yellow colour changing to red appears. A solution in  $Ac_2O$  acquires on the addition of concentrated  $H_2SO_4$  a violet-red colour tending to blue. It yields an amorphous cholesteride, m.p. 260°C. The Ca salt from MeOH forms flat prisms m.p. 255°–258°C. Hot 5 per cent.  $H_2SO_4$  resolves the saponin very slowly into sapogenin, glucose and glucuronic acid. The sapogenin is separated by boiling petroleum ether into  $\alpha$ -sapogenin,  $C_{33}H_{52}O_6$ , granular warty crusts m.p. 150°C., and  $\beta$ -sapogenin,  $C_{33}H_{52}O_7$ , colourless powder m.p. 225°–228°C. Alcoholic KOH hydrolyzes sapogenin into tiglic acid and 2 alcohols, one easily soluble in EtOH m.p. above 300°C., the other difficultly soluble in EtOH m.p. 298°C.

**Urease, Preparation of, in Powder Form.** M. Jacoby and — Sugga. (*Biochem. Zeit.*, 1915, 69, 116; *Chem. Abstr.*, 1915, 9, 1492.) Soya-bean meal is defatted with successive quantities of petroleum ether. The fat-free powder freed from the solvent is then macerated with 5 times its weight of water for 16 to 24 hours at 0°C. After filtration or centrifugation, the liquid is evaporated at the normal temperature in a current of dry air and the residue is powdered. The powder is slowly and incompletely soluble in water. Treatment of the soya meal with 70 per cent. EtOH give a water-insoluble dry extract of about the

same activity as the above. Heating to 70°C. destroys a greater part of the activity of the urease, while at 60°C. very little effect is produced. Trypsin digestion has no influence upon its activity. The action of papayotin, even at 60°C., has no influence upon the activity, but if the urease-papayotin mixture is dialyzed, there is a considerable decrease in the activity of the urease action. This may be due to the loss of auxosubstances, though the urease preparation itself was not changed by dialysis.

**Urease from Soya Beans, Activation of, by Human Serum.** R. Neumann. (*Biochem. Zeit.*, 1915, 69, 134-40; *Chem. Abstr.*, 1915, 9.) Human serum increases the urea-splitting activity of soya urease about 8 times. A quantitative difference in this auxo-ureatic action could not be established for sera of different diseases, the amount being fairly constant in all cases. Fluid from a pleural puncture had the same effect as serum. That from lumbar puncture is not as active, the greatest activity being found in a case of tubercular meningitis. ●

## GUMS, OLEORESINS AND RESINS

**Acacia in Tragacanth, Detection of.** Gilg. (*Drugg. Circ.*, 1914, 58, 667.) One Gm. of tragacanth is mixed with 50 c.c. of water and 2 Gm. of tincture of guaiacum, and the mixture is allowed to stand for 3 hours. When acacia is present, a blue colour will be produced in the mixture by the oxydases of the gum.

**Benzoin, Siamese, New Crystalline Constituent of.** F. Reinitzer. (*Arch. Pharm.*, 1914, 252, 341; *J.S.C.I.*, 1915, 34, 681.) Siamese gum benzoin, according to Luedy (*Y.B.*, 1894, 171), consists mainly of a mixture of two benzoic esters, the alcohols being benzoiresinol,  $C_{16}H_{26}O_2$ , and siaresinotannol,  $C_{12}H_{14}O_3$ . A new colourless crystalline benzoate of a previously unknown resin-alcohol, for which the name lubanol is proposed, has been isolated.

**Daniella thurifera, Oleoresin of.** (*Bull. Imp. Inst.*, 1915, 13, 44.) The tree, also known as *Paradaniella oliveri*, is widely distributed through western tropical Africa. The oleoresin was at one time exported under the name of "Illurin balsam," and is stated to be used for adulterating copaiba. Two samples of the oleoresin have been examined at the Imperial Institute.

One from the Gold Coast gave 33 per cent. of volatile oil: sp.g. 0.927;  $\alpha_D + 9.37'$  at  $24^\circ\text{C}$ .;  $\eta_D$  1,509; b.p. 253–271. The other specimen from Nigeria gave 35 per cent. of oil: sp.g. 0.923;  $\alpha_D + 19.5'$  at  $20^\circ\text{C}$ .;  $\eta_D$  1,506. The oleoresin has not yet been examined therapeutically.

**Dipterocarpus crinitus, Oleoresin from.** (*Bull. Imp. Inst.*, 1915, 13, 42.) The tree yielding the oleoresin grows abundantly at Selangor, Straits Settlements. The oleoresin known as "wood oil" or "Kruing sap" is an opaque, very viscous greyish-white liquid, with an aromatic but slightly sour odour. It yielded only 19.5 per cent. of a yellow essential oil to steam distillation similar in characters and reactions to that obtained from gurjun balsam which generally gives over 50 per cent. of essential oil.

**Elemi, African, Cameroon, Gum.** K. Dietrich. (*Pharm. Post*, 46, 808.) Two authentic samples of this gum were examined. The first sample probably originated from *Canarium schweinfurthii*, was hard, had m.p. about  $110^\circ\text{--}112^\circ\text{C}$ ., acid value 27.45–27.48, saponification value (hot) 76.66–76.75. Of the unevaporated  $\text{Et}_2\text{O}$  extract the acid value was 26.04 and saponification value 49.18. The gum contained afamyrin, resin, resin acid, bitter principle and a little over 1 per cent. of essential oil. The second sample probably originated from *C. mansfeldianum*, was somewhat hard, white, had m.p.  $108^\circ\text{C}$ ., acid value 27.77–20.60, saponification value (hot) 63.03–54.02. The  $\text{Et}_2\text{O}$  purified resin had an acid value 23.63–23.83, saponification value (hot) 51.55–52.66. Its constituents were resin, resin acid, bitter principle and 3.26 per cent. of essential oil.

**Kauri Gum.** J. B. Aitken. (*Pharm. J.*, 1915 [4], 40, 550.) An interesting description of the Kauri pine, *Dammara australis*; and the fossil gum kauri, which is an important article of export from New Zealand. A full description of the method of seeking the buried gum and of its commerce are given.

**Mastic, Artificial and Distilled.** K. Dieterich. (*Pharm. Zeit.*, 1914, 59, 912; *Chem. Abstr.*, 1915, 9, 1092.) Three samples were examined, two of artificial origin and the third a residue obtained in the distillation of true mastic. (1) Artificial mastic (Worlee-Hamburg), small brownish yellow deformed grains adhering together on slight warming, in appearance readily distinguished from true mastic. Solutions in  $\text{C}_6\text{H}_6$  and  $\text{C}_2\text{H}_5\text{OH}$



are nearly clear ; in  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ , acetone, 96 per cent.  $\text{AcOH}$ ,  $\text{CS}_2$  and turpentine slightly turbid ; in 90 and 96 per cent.  $\text{EtOH}$ ,  $\text{MeOH}$ , benzin strongly turbid ; in petroleum ether very strongly turbid. It is soluble in paraffin and fatty oils, thus possessing solubilities similar to true mastic. The Storch-Morawski reaction develops a red-violet instead of a yellow colour as in the case of true mastic. If 0.5 Gm. is dissolved in 5 c.c. of  $\text{EtOH}$  and 5 c.c.  $\text{HCl}$  is added to the cold solution, a white turbidity results in the case of true mastic, while the artificial product yields in addition a very beautiful violet colour growing more intense on standing. Acid value, 22.81–22.94 ; saponification value, 49.92–50.47 ; ash, 0.15 per cent. (2) Artificial mastic "Albertol," light brown, brittle masses like colophony and possessing an odour like lilies of the valley. Yields clear solutions with  $\text{Et}_2\text{O}$ ,  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ , fatty oils and turpentine ; partially soluble in 90 and 96 per cent.  $\text{EtOH}$  and benzin ; almost completely soluble in hot 96 per cent.  $\text{AcOH}$ . The Storch-Morawski test yields a transient violet colour. On dissolving a small amount in 3 c.c. of  $\text{CHCl}_3$  and adding thereto 3 c.c. of  $\text{H}_2\text{SO}_4$  the latter becomes red, the  $\text{CHCl}_3$  reddish. The  $\text{EtOH}$  solution gives with  $\text{HCl}$  a white turbidity but no colour reaction like the Hamburg product. Acid value, 22.40 ; saponification value, 102.2–103.6, m.p. 56–58°. (3) Distilled mastic (Sachse & Co.—Leipzig) occurs in odourless, colourless, light brown masses like colophony, and behaves like sandarac on chewing. Completely soluble in  $\text{C}_6\text{H}_6$ ,  $\text{CHCl}_3$ ,  $\text{Et}_2\text{O}$ , amyl alcohol, turpentine and fatty oils ; partially soluble in  $\text{EtOH}$ ,  $\text{AcOH}$ , acetone, benzin, petroleum ether and  $\text{MeOH}$ . Storch-Morawski test yields an evanescent violet colour. With the  $\text{CHCl}_3$ - $\text{H}_2\text{SO}_4$  test the acid becomes red, the  $\text{CHCl}_3$  reddish. The  $\text{EtOH}$  solution gives no colour with  $\text{HCl}$ . Acid value, 47.68 ; saponification value, 64.54.

***Pinus clausa*, Oleoresin of.** A. W. Schorger. (*J. Ind. Eng. Chem.*, 1915, 7, 321.) *Pinus clausa*, the sand pine, is confined to the State of Florida. Its oleoresin is very impure as collected, containing water and dirt. In the specimen examined the amount of volatile oil was 18.9 per cent. and of resin 72.3 per cent. The oil contained laevo- $\alpha$ -pinene, 10 per cent. ; camphene, 10 per cent. ; and laevo- $\beta$ -pinene, 75 per cent. The resin consists mainly of abietic acid.

***Pinus halapensis*, Oleoresin and Essential Oil of.** E. Tsakalotos. (*J. Pharm. Chim.*, 1915, 11, 70.) The oleoresin de-

scribed was derived from Aleppo pines growing in Attica. It yielded 21.4 per cent. of essential oil and 70.8 per cent. of rosin. The essential oil consisted almost entirely of pure dextro-pinene. It differed scarcely at all from the oil obtained from the oleoresin of the same species collected at different periods in other localities.

**Podophyllum Resin, Determination of, in Liquid Extract of Podophyllum.** W. M. Jenkins. (*J. Ind. Eng. Chem.*, 1914, 6, 671.) Measure 5 c.c. of fluid extract podophyllum into a separator, add 5 c.c. of EtOH, 10 c.c. of  $\text{CHCl}_3$ , and 10 c.c. of acidulated water containing 0.6 per cent. HCl (2 c.c. HCl in 100 c.c. water). Shake and allow the mixture to separate. Draw off the lower layer into another separator. Repeat the extraction twice, using 15 c.c. of a mixture of 1 part of EtOH and 2 parts of  $\text{CHCl}_3$  each time, and add these extractions to the first. Shake the combined extractions with 10 c.c. of the acidulated water, and allow the mixture to separate. Draw off the lower layer into a tared flask, and repeat the extraction twice, using 15 c.c. of the alcohol-chloroform mixture each time. Evaporate the combined extractions and dry the residue to constant weight at 100°C.

In assaying the drug, 10 Gm. in a No. 60 powder is placed in an Erlenmeyer flask, and 25 c.c. of EtOH is added and heated, under a reflux tube condenser at 80°C. for 3 hours. The contents of the flask are then transferred to a small percolator and washed with alcohol until about 50 c.c. of percolate is obtained. When cooled to room temperature, the solution is made up to exactly 50 c.c. Of this solution 10 c.c., representing 2 Gm. of the drug, is used for assay, which is carried out as described for the fluid extract, with the exception of the addition of the 5 c.c. of EtOH, which is omitted.

The method can also be applied to the assay of solid and powdered extracts of podophyllum by dissolving weighed quantities of the extracts in sufficient EtOH to render the solutions of about the same strength as fluid extract.

**Stick Lac, New Alcohol from.** A. Gascard. (*Comptes rend.*, 1914, 159, 258.) Resin and wax are removed from stick lac by boiling with EtOH 95 per cent. The insoluble residue consists of woody impurities and the bodies of the lac insect *Tachardia lacca* filled with colouring matter. The insect portion is separated and boiled with  $\text{C}_6\text{H}_6$ . This removes a waxy substance which, when purified by recrystallization from benzene,

melts at  $94^{\circ}\text{C}$ . It is the ester of a new alcohol; insoluble in  $\text{EtOH}$ , in  $\text{Et}_2\text{O}$ , and in  $\text{HC}_2\text{H}_3\text{O}_2$ ; soluble in  $\text{CHCl}_3$  and in  $\text{C}_6\text{H}_6$ . When saponified with  $\text{KOH}$ , the new alcohol, *laccero*,  $\text{C}_{33}\text{H}_{66}\text{O}$  is liberated; forming lozenge-shaped crystals, m.p.  $95^{\circ}$ – $96^{\circ}\text{C}$ ., sparingly soluble in the cold, in  $\text{EtOH}$ ,  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$  and  $\text{C}_6\text{H}_6$ ; readily soluble on warming. The acid obtained in this saponification *lacceroic acid*  $\text{C}_{33}\text{H}_{64}\text{O}_2$  is closely allied to psyllostearic acid, obtained by Sundwick from *Psylla alni*.

**Tragacanthins.** T. v. Fellenberg. (*Mitt. Lebensm. Hyg.*, 1914, 5, 256–9; *Chem. Abstr.*, 1915, 9, 624.) The water soluble portion of gum tragacanth does not contain a  $\text{MeOH}$  group, but the insoluble part, known as bassorin, when treated with  $\text{NaOH}$ , dissolves and yields a solution which, acidified with  $\text{HCl}$  and distilled, gives  $\text{MeOH}$ . There is present in cellulose, starch, and ash-free bassorin 5.38 per cent.  $\text{MeOH}$ . When bassorin is split up by  $\text{NaOH}$ , it yields, besides  $\text{MeOH}$ , a weak acid, bassoric acid, previously termed oxybassorin, and given the formula  $(\text{C}_{11}\text{H}_{20}\text{O}_{10})_2\text{O}$ , to correspond with the usually accepted formula  $(\text{C}_{11}\text{H}_{20}\text{O}_{10})_n$  for bassorin. If the radicle be doubled, and the  $\text{MeO}$  group substituted for  $\eta$ , we have a theoretical  $\text{MeOH}$  content of 5.13 per cent.; that experimentally found is 5.38 per cent. Bassoric acid can be titrated with  $\text{NaOH}$ , using phenolphthalein. It does not liberate  $\text{CO}_2$  from the alkali earth carbonates. With  $\text{EtOH}$  and many electrolytes it yields a jelly or in dilute solution a flocculent precipitate. Bassoric acid is coagulated by  $\text{Ba}(\text{OH})_2$ ,  $\text{BaCl}_2$ ,  $\text{AlCl}_3$ ,  $\text{FeCl}_3$ ,  $\text{AgNO}_3$ ,  $(\text{PbNO}_3)_2$ , both neutral and basic lead acetate;  $\text{ZnSO}_4$  produces an opalescence, but no precipitate. The free acid is not coagulated by mineral acids, alkali salts,  $\text{CaCl}_2$ ,  $\text{SrCl}_2$ ,  $\text{MgCl}_2$ ,  $\text{FeSO}_4$ ,  $\text{MnCl}_2$ ,  $\text{Co}(\text{NO}_3)_2$ ,  $\text{NiSO}_4$ ,  $\text{CdCl}_2$ , or  $\text{HgCl}_2$ . Most of these precipitate the  $\text{Na}$  salt, but  $\text{MgCl}_2$ ,  $\text{CaCl}_2$ , and  $\text{HgCl}_2$  do not. Basic dyes coagulate bassoric acid, but tannin and albumin do not. In this respect it differs from pectic acid.

## INORGANIC CHEMISTRY

**Airol, Valuation of.** (*Supplement to Ph. Ned. IV.*; *J. Pharm. Chim.*, 1915, 11, 240.) Airol or bismuth oxyiodogallate should contain at least 20 per cent. of  $\text{I}$ . when determined as follows. Airol 0.5 Gm. is heated with  $\text{NaOH}$  solution 10 c.c. After

cooling,  $\text{HNO}_3$  20 c.c. is added and 20 c.c. of N/10  $\text{AgNO}_3$  solution. The mixture is boiled for about 30 minutes, and cooled. Distilled water, 100 c.c., is added, then 5 drops of iron alum, and the excess of  $\text{AgNO}_3$  is determined by means of N/10  $\text{AmCNS}$  solution. No more than 12 c.c. of the latter should be needful to give a pink colour.

**Ammonia Distillation Flask, New.** G. W e m p e. (*Z. angew. Chem.*, 1914, 27, I, 624; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 257.) The flask is a modification of that of Abderhalden. (See fig.) Capacity of *A* = about 400 c.c., *B* about 150 c.c., *C* about 200 c.c. A condenser is connected to *C*. Funnel *B* serves for introduction of  $\text{NaOH}$  and obviates any loss that may occur in forms requiring connection after addition of alkali; such loss amounting to 0.2 per cent. or more of  $\text{NH}_3$ . During the distillation a slow current of air enters through *B*.



**Ammonia, Determination of, by Nesslerizing, Standard Colour for.** A. R. Rose and Katherine R. Coleman. (*Biochem. Bull.*, 3, 407; *Chem. Abstr.*, 1914, 8, 3801.) A 5 per cent. solution of chloroplatinic acid to which has been added half its volume of  $\text{HCl}$  solution containing 1.2 per cent. of  $\text{CoCl}_2$  is used as a standard colour instead of the nesslerized  $\text{Am}_2\text{SO}_4$  solution. The modification of the method is very accurate for solutions containing 0.4–2 Mgm. N per c.c. The colour is allowed to develop for 15 minutes and the readings are made within 10 minutes. For solutions very low in N, the stronger standard should be diluted, but its N equivalent must be obtained by comparing it with a nesslerized standard  $\text{NH}_3$  solution as its N equivalent is not proportionate to the dilution.

**Ammonia, Sensitive Reagent for.** Sara Graves. (*J. Amer. Chem. Soc.*, 1915, 37, 1171.) Saturated aqueous  $\text{HgCl}_2$  solution, 50 c.c., is mixed with  $\text{NaCl}$ , 15 Gm.; saturated solution of  $\text{Li}_2\text{CO}_3$ , 35 c.c., and distilled water 65 c.c. are added. This will give an evident precipitate with a dilution of 1 of  $\text{Am}_2\text{SO}_4$  in 1,000,000.

**Arsenic Compounds, Biochemical Decomposition of.** H. H u s s. (*Zeit. Hyg.*, 1914, 76, 361; *Chem. Abstr.*, 1914, 8, 3807.) There are a very few fungi that are capable of separating As from

its compounds and volatilizing it. These As fungi are called *Penicillium brevicaulis* and *Actinomyces* sp. All As compounds can, without exception, be decomposed if the conditions are favourable. The most favourable conditions are  $O_2$ , dampness and a suitable medium. The absence of one of these factors hinders the growth of the fungus and consequently the volatilization of the As. The water-insoluble As compounds are much more slowly decomposed than those soluble in water. The mass of volatilized As is proportional to the reacting ability of the fungus and the  $H_2O$  solubility of the given As compound. The As-containing gas liberated possesses little toxicity.

**Arsenic, Detection and Determination of, in Medicinal Organic Compounds.** L. Barthe. (*Bull. Soc. Pharm. Bordeaux; Répertoire de Pharm.*, 1915, 27, 144.) As is well known As in intimate molecular combination, such as occurs in the modern therapeutic preparations, does not respond to the ordinary direct reagents. Although Bougault's reagent, and especially when rendered more sensitive by the addition of iodine, will give reactions when heated with sodium methylarsenate, atoxyl, arsacetin, hectine, enesol, salvarsan, and neosalvarsan, and affords the odour of cacodyl with sodium cacodylate, it also gives reduction precipitates and colours with so many other metallic substances, that its value is not great in this case. The same defect applies to Bresannin's test which depends on the insolubility of  $AsI_3$  in  $H_2SO_4$  or  $HCl$ , the substance being dissolved in the acid and treated with  $KI$  solution. These methods are not quantitative. The author treats from 0.40 to 0.5 Gm. of the material with 25 to 30 c.c. of strong  $H_2SO_4$ , heating the mixture over the naked flame, in a flask with a funnel condenser. The organic molecule is then sufficiently broken up, and the As may be determined in the majority of cases, as  $As_2S_3$ . Sometimes the blackish acid residue will need further purification. In the case of antipyrine methylarsenate and cacodylate the antipyrine should be liberated with  $NaOH$  and shaken out with  $CHCl_3$ , and the alkali cacodylate or methylarsenate are then treated as above with  $H_2SO_4$ .

**Arsenious Acid and Sodium Arsenate, Determination of, in Granules.** M. François and E. Lasausse. (*J. Pharm. Chim.*, 1915, 11, 226.)  $As_2O_3$ .—At least 50 granules are counted, or, if possible, 100; the weight is determined to give the mean weight per granule. These are transferred to a conical flask and

heated on the boiling water-bath under a hood for 3 hours with 20 c.c. of  $\text{HNO}_3$  sp.g. 1.357. The residual clear liquid may then show a few crystals of mucic acid. Without regarding these, 20 c.c. of water is added, and a fragment of litmus paper, then gradually  $\text{AmOH}$  until a markedly alkaline reaction is given. If necessary the liquid is filtered and the filter washed. Twenty c.c. of magnesium mixture, made with  $\text{MgCl}_2$ , is added, and 20 c.c. of  $\text{AmOH}$  sp.g. 0.924. The mixture is then set aside for 3 days. The precipitate is washed by decantation with solution of  $\text{AmOH}$  1 and water 3; collected on a counterpoised filter; washed, dried at  $100^\circ\text{C}$ . for 10 hours and weighed. The dry salt has the formula  $[\text{AsO}_4(\text{NH}_4)\text{Mg}]_2 \text{H}_2\text{O}$ ; its molecular weight is  $190 = 99$  of  $\text{As}_2\text{O}_3$ .

*Sodium Arsenate Granules.*—The method of procedure is precisely the same as for  $\text{As}_2\text{O}_3$  granules, but the number of granules counted should be at least 100, and, if possible, 200. The results obtained should be calculated into the official sodium arsenate  $\text{Na}_2\text{HAsO}_4 + 7\text{H}_2\text{O}$ , the molecular weight being 312, corresponding to 190 of  $[\text{AsO}_4(\text{NH}_4)\text{Mg}]_2 \text{H}_2\text{O}$ .

**Arsenium Iodide and its Solution.** G. and J. Languépin. (*Bull. Synd. Pharm. Charente; Répertoire Pharm.*, 1915, 27, 91.) Arsenium iodide may be easily prepared in the pharmacy. Metallic As, 20 Gm., is suspended in nearly boiling water 400 or 500 c.c.; I, 10 Gm., is added and the temperature maintained. A change of colour ensues; the liquid is boiled for 10 minutes, filtered, and evaporated to dryness on the water-bath. The  $\text{AsI}_3$  thus obtained may be redissolved in  $\text{CHCl}_3$ . On evaporating the solvent, it forms red hexagonal scales. It is readily decomposed;  $\text{EtOH}$  and  $\text{Et}_2\text{O}$  cause its dissociation.

Solution of  $\text{AsI}_3$  may be prepared from  $\text{As}_2\text{O}_3$  and KI. Thus by boiling a solution of 2.172 Gm. of  $\text{As}_2\text{O}_3$  with 10.93 Gm. of pure KI in 1,000 c.c. of water, a solution of 10 Gm. of  $\text{AsI}_3$  is obtained. Since the official KI of the French Codex contains only 98 per cent. of the pure salt, 11.152 Gm. of the official KI must be used to obtain a 1:100 solution of  $\text{AsI}_3$ .

**Associated Precipitation for Detection and Determination of Small Quantities in Complex Mixtures.** G. Mellière. (*Annales Chim. analyt.*, 1915, 20, 73.) The method of associated precipitation is finding more general application on analysis for the isolation of small amounts of many substances which occur

in presence of a preponderant excess of other constituents. This precipitation, which in many instances has accounted for the overlooking of traces during an analysis, may be turned to account for the detection of small quantities. Instances of associated precipitation are familiar to those engaged in water analysis and may be instanced by the separation of the less common constituents, notably As and Mn, from spring waters when exposed to the air. It has also been employed for the precipitation of infinitesimal quantities of metals from water, by means of  $\text{Ba}(\text{OH})_2$ , or by the addition of a little  $\text{CuSO}_4$  for the detection of minute quantities of Hg, which, when precipitated as  $\text{CuS}$ , carries down with it the Hg in association. The author employs the method for the toxicological detection of Pb. The suspected liquid is treated with 0.25 Gm. of electrolytic  $\text{CuSO}_4$  for each litre, treated with 10 c.c. of  $\text{HCl}$ , all sediment being included in the mixture, and  $\text{H}_2\text{S}$  passed through the mixture. If the liquid has stood for some time in a vessel before analysis, the above  $\text{HCl}$  should be used to rinse this, for all the Pb present may be contained in the deposit.  $\text{H}_2\text{S}$  is then passed through the acid liquid in the cold, without agitation. After standing for 24 hours the precipitate is collected on a filter or on a filter pump with asbestos pulp. After washing with tepid water, a few drops of  $\text{HNO}_3$  are poured over the filter, which is then washed, and the acid filtrate is evaporated to dryness. The residue is ignited to decompose the  $\text{Cu}(\text{NO}_3)_2$ , again treated with  $\text{HNO}_3$  and submitted to electrolysis in presence of not more than one-fifth its volume of  $\text{HNO}_3$  with a current of 2 volts and less than 0.2 ampères; the temperature of the bath should not exceed  $40^\circ\text{C}$ . The electrodes should be small Pt spirals weighing from 2 to 4 Gm. on which the deposits of  $\text{PbO}_2$  can be readily weighed. Obviously care must be taken to ensure the purity of all the reagents employed.

**Bettendorf's Reagent and its Modification.** Vanino and Hartwagner. (*Arch. Pharm.; Drugg. Circ.*, 1915, 59, 17.) The sensitiveness of the various modifications has been compared.

*Reagent of the German Pharmacopœia.*  $\text{SnCl}_2$ , 5; is mixed with  $\text{HCl}$ , 1; and into the mixture dry  $\text{HCl}$  gas is conducted until the mixture is saturated. *Immendörfer-Kornthal's Reagent* is prepared by dissolving  $\text{SnCl}_2$ , 1, in  $\text{HCl}$  30 per cent. 3;

and adding to  $\text{H}_2\text{SO}_4$ , 1; keeping the mixture cool. This mixture is said to have many advantages over others, because it is colourless when prepared from reagents absolutely free from arsenic, and because its reducing power is so strong that heating is not necessary. *Warnecke's Reagent* consists of a solution of crystalline  $\text{SnCl}_2$ , 1;  $\text{HCl}$ , 38 to 40 per cent. (sp.g. 1.19 to 1.20) 2. This reagent gives as accurate results as those obtained by Marsh's arsenic test. *Lobello's Reagent* is made by shaking one kilo of crystalline  $\text{SnCl}_2$ , 1; with  $\text{HCl}$  (sp.g. 1.19) 1. *Moberger's Reagent* is based upon the fact that the delicacy of Bettendorf's reagent is not reduced when less stannous chloride is used. Crystalline  $\text{SnCl}_2$ , 1; in  $\text{HCl}$  (sp.g. 1.19) 5.

*Winkler's Reagent* is a solution of  $\text{SnCl}_2$ , 100 Gm. 36 to 38 per cent.  $\text{HCl}$  to obtain 1,000 c.c. *Farraro and Carobbio's Reagent* is made from metallic tin instead of from  $\text{SnCl}_2$ . The arsenic test is carried out by adding to the liquid under examination 0.02 to 0.04 Gm. of metallic tin followed by 10 to 12 drops of concentrated  $\text{HCl}$ . The sensibility of this reagent is claimed to be as great as that of Bettendorf's original reagent. *De Jong's Reagent* contains  $\text{Et}_2\text{O}$  as a solvent for  $\text{SnCl}_2$ .  $\text{SnCl}_2$ , 25 Gm. is shaken with  $\text{Et}_2\text{O}$  100 c.c., and when most of the salt is dissolved, sufficient  $\text{HCl}$  is added to the  $\text{Et}_2\text{O}$  solution to obtain a practically clear mixture. The As test is carried out by adding to the liquid under examination 5 c.c. of  $\text{HCl}$ , then 5 c.c. of the reagent, shaking the mixture and allowing it to stand for one minute in a water-bath at  $40^\circ$ . In the presence of As a brownish-red ring will be formed at the zone of contact of the ethereal layer and of the aqueous liquid. By applying these various tests to arsenious and arsenic solutions of varying strength, the authors arrive at the following conclusions: Either one of the methods can be used for detecting pentavalent arsenic. By the methods of Ferraro and Carobbio and by that of de Jong as little as 0.03 Mgm. of pentavalent arsenic can be detected. The most sensitive reagent for trivalent arsenic is de Jong's reagent, by use of which the presence of 0.0015 Mgm. of arsenious acid can be shown. By the method of the German Pharmacopœia 0.006 Mgm. of pentavalent arsenic can be detected.

Finally, the authors conclude that for ordinary use the reagents of Warnecke, Moberger or Winkler are quite satisfactory, since these can be prepared by a very simple process, and since by them as little as 0.015 Mgm. of either pentavalent or trivalent arsenic can be detected.



**Boric Acid, Determination of Small Quantities of.** G. Halphen. (*Annales des Falsifications*, 1915, 8, 1; *J.S.C.I.*, 1915, 34, 278.)  $\text{H}_3\text{BO}_3$  is separated by distillation as methyl borate, which is received in 0.2 c.c. of N/NaOH solution; the alkaline solution is evaporated to dryness and the residue dissolved in 1 c.c. of water and 2 c.c. of HCl (sp.g. 1.162). Comparison solutions are prepared at the same time, containing definite, successively increasing quantities of  $\text{H}_3\text{BO}_3$ . To each of the tubes is added 1 c.c. of a solution of turmeric in ethyl acetate, and the red colorations which develop are compared after the lapse of 50 minutes.

**Calomel in Tablets, Method for Determining.** J. W. Marsden and O. E. Cushman. (*Amer. J. Pharm.*, 1914, 86, 511.) After trial of the usual methods for the determination of HgCl the authors devised the following process as being most suitable and accurate in the case of tablets. It was subsequently found that Kohn and Ostersetzer had published a practically identical method, using  $\text{H}_2\text{O}_2$  to reduce the HgCl. The tablets, in amount corresponding to 0.2 to 1.0 Gm. of HgCl, are first disintegrated in about 30 c.c. of water, made acid with  $\text{HNO}_3$  to drive off the  $\text{CO}_2$  from the  $\text{NaHCO}_3$ , which is often used as a filler, and  $\text{Na}_2\text{O}_2$  added, a little at a time, with stirring, until grey metallic Hg separates out. About 1 Gm. of  $\text{Na}_2\text{O}_2$  is added in excess. After heating for a very few minutes, the precipitated Hg is filtered on to a Gooch crucible and washed with water. The filtrate is strongly acidified with  $\text{HNO}_3$ , N/10  $\text{AgNO}_3$  added in excess, and the solution then cooled and agitated, causing the precipitate to aggregate. The excess of N/10  $\text{AgNO}_3$  is then titrated with N/10 KCNS, using ferric alum as indicator.

**Calomel in Tablets, Determination of.** R. I. Grantham. (*J. Amer. Pharm. Assoc.*, 1915, 4, 441.) The following method gives satisfactory results in the determination of HgCl. Three Cgm. of calomel or an equivalent amount of the powdered tablets is transferred to a 4-ounce Erlenmeyer flask, mixed with 0.5 Gm. of  $\text{KClO}_3$  and 15 to 20 c.c. of 10 per cent. HCl. The mixture is digested on a steam-bath for 15 minutes and then filtered into a large beaker, the filter washed well with water and the filtrate made alkaline with  $\text{AmOH}$ . After the addition of a large excess of acetic acid and 1 to 3 Gm. of  $\text{K}_2\text{C}_2\text{O}_4$  to the filtrate, the mixture is boiled, stirring constantly

in order to prevent bumping. After allowing to settle it is filtered. The precipitate is washed 3 times with hot water by decantation, transferred to a filter and washed with hot water until free from Cl. The filtrate and wash-water are then heated to about  $80^{\circ}\text{C}$ . and the Hg is precipitated with  $\text{H}_2\text{S}$  and weighed as  $\text{HgS}$ .

**Colloidal Silver Preparations, Determination of Ag in.** P. W. D a n c k w o r t. (*Archiv. Pharm.*, 1914, 252, 497.) One Gm. of the substance is treated in an Erlenmeyer flask with 10 c.c. of cold water; a mixture of perhydrol, 5 c.c., and  $\text{HNO}_3$ , 25 per cent., 15 c.c. is then slowly added with constant agitation. The whole is then warmed on the water-bath for 30 to 45 minutes, with frequent agitation until only about 5 c.c. of liquid remains. This is diluted with water up to 100 c.c. and titrated with N/10 AmCNS and iron alum indicator. The method answers well for protargol and those preparations which contain no appreciable amount of Cl. For collargol and other compounds which contain Cl, the method of Lehmann gives good results.

**Compound Licorice Powder, Determination of S in.** M a r g a r e t J C r o s b i e and W. H. Gibson. (*Chem. & Drugg.*, 1914, 85, 72.) Owing to the presence of organic matter the usual method of oxidation of S to  $\text{H}_2\text{SO}_4$  by  $\text{HNO}_3$  will easily give low results, about 4 per cent. A sample of compound liquorice powder which was alleged to contain only 4 per cent. of S, and found by analysis by the  $\text{HNO}_3$  method to give that result. Extraction by  $\text{CS}_2$ , however, gave 7 per cent. of S, showing that the first method was faulty. The following modification of the oxidation method was finally found to give the correct percentage of sulphur—namely, 8 per cent. One 4 Gm. of the sample is treated with 50 c.c. of 1.5  $\text{HNO}_3$  and a little powdered  $\text{KClO}_3$ , and evaporated to dryness. 0.5 Gm. of powdered  $\text{KClO}_3$  and 5 c.c. of  $\text{HCl}$  are added, and the evaporation repeated. A second quantity of  $\text{KClO}_3$  and  $\text{HCl}$  is added and again evaporated. The residue is then treated with 100 c.c. of distilled water and boiled, and the  $\text{H}_2\text{SO}_4$  precipitated with  $\text{BaCl}_2$  solution. It is preferable to allow the precipitate to stand overnight, when it is filtered and weighed as  $\text{BaSO}_4$ .

**Copper, New Test for, with  $\alpha$  Amino-caproic Acid.** W. G. L y l e, L. J. C u r t m a n, and J. T. W. M a r s h a l l. (*J. Amer. Chem. Soc.*, 1915, 37, 1471.) An aqueous solution of nor-

mal amino-caproic acid is an exceedingly sensitive reagent for the detection of Cu. With this reagent 0.004 Mgm. of Cu may be detected with certainty. Hg and Zn are the only other common metals which yield, under the conditions specified, a precipitate with the reagent. The interference of the former may be overcome by the addition of NaCl, the latter may be prevented from precipitating by adjusting the acidity of the solution. The reagent is more specific for copper than any of the other reagents heretofore proposed; and possesses an advantage over the  $K_4FeCy_6$  test in that small quantities of Fe do not interfere with its use. The reagent consists of 0.67 Gm. of amino-caproic acid dissolved in 100 c.c. of water by aid of heat, then cooled and filtered. To prevent the formation of free mineral acid in the solution to be tested a 4 per cent. solution of  $AmC_2H_3O_2$  was added first. One c.c. of the neutral solution to be tested is treated with 1 c.c. of 40 per cent.  $AmC_2H_3O_2$  solution and 1 c.c. of the amino-caproic acid solution. With considerable amounts of Cu a greyish-blue precipitate is formed. With 0.000004 Gm. of Cu a distinct reaction occurs in 5 minutes. Details of special manipulations necessary in presence of large quantities of other metals are given.

**Copper, Volumetric Determination of, with Sodium Nitroprusside.** G. Zucchari. (*Boll. Chim. Farm.*, 1914; *J. Pharm. Chim.*, 1915, 11, 187.) A N/10 solution containing 14.496 Gm. of the salt in 1,000 c.c. may be used; or if preferred one containing 46.866 Gm. in the litre. Of the latter 1 c.c. = 0.01 Gm. of Cu. The end reaction is obtained by means of an indicator of an alkali sulphide, by spotting out. The usual impurities in commercial Cu salts do not react with nitroprusside. If ferrous Fe is present in quantity exceeding 2 Gm. (per litre) it must be oxidized. In presence of Cd or Ni titration should be performed in dilute solutions.

**Dental Cements, Germicidal Efficiency of.** P. Poetschke. (*J. Ind. Eng. Chem.*, 1915, 7, 195.) Dental cements used on account of their supposed bactericidal properties are known as "Copper cements," or "Copper oxyphosphates." Many types are on the market as white, red, black, and varicoloured powders. These are massed for use with a little  $H_3PO_4$  modified by the addition of  $Al_2(OH)_6$ . Sometimes other metals, such as Fe or Ni, are added.  $Al_2(OH)_6$  is used as a modifier in order to control the reaction of the liquid on the powder when the two are combined

by spatulation. White copper cement powder consists of calcined  $\text{ZnO}$ ,  $\text{MgO}$ , and  $\text{Bi}_2\text{O}_3$ , the Cu being added in the form of  $\text{Cu}_2\text{I}_2$ ,  $\text{CuHPO}_4$ ,  $\text{CuSiO}_3$ , or other light-coloured copper salts. Black copper cement powder consists of either  $\text{CuO}$  and  $\text{CoO}$ , or  $\text{CuO}$  and  $\text{ZnO}$  with or without other oxides such as  $\text{MgO}$  and  $\text{Bi}_2\text{O}_3$ . Red copper cement powder consists essentially of  $\text{ZnO}$  and  $\text{Cu}_2\text{O}$ , other oxides such as magnesia and bismuth being occasionally present. Red pigments are also added to improve the colour, which is not a deception, provided sufficient  $\text{Cu}_2\text{O}$  is used to give the cement mass the requisite germicidal power. The varicououred cement powders consist essentially of  $\text{ZnO}$ ,  $\text{MgO}$ , and  $\text{Bi}_2\text{O}_3$ , with a small proportion of light-coloured copper compounds such as  $\text{CuSiO}_3$ ,  $\text{CuHPO}_4$ , or other light-coloured copper salts. Pigments are added to obtain the various shades. The results of the examination of these cements and their respective ingredients, from the point of germicidal efficiency, are summarized as follows.  $\text{Cu}_2\text{O}$ ,  $\text{CuO}$  and  $\text{ZnO}$  have marked germicidal properties. The addition of  $\text{Cu}_2\text{O}$ ,  $\text{CuHPO}_4$  and  $\text{Cu}_2\text{I}_2$ , to  $\text{ZnO}$  enhances the bactericidal properties of the latter. The addition of varying amounts of  $\text{Cu}_2\text{I}_2$  to a "copper-free" dental cement shows that the germicidal efficiency is increased in proportion to the quantity added. Commercial "copper cements" show wide differences in germicidal efficiency, the colour of the cement having no relation to its bactericidal properties. Tests for germicidal properties of dental cements in "plastic" or "set" condition are unsatisfactory if such mixes are placed in inoculated bouillon or agar-agar, owing to the difference in chemical composition of the nutrient culture media and the saliva. Visual tests for inhibition of bacterial growth are purely qualitative tests and fail to demonstrate the comparative germicidal efficiency of these materials. The comparative germicidal efficiency of "copper cements" can be ascertained only by methods which determine the number of living organisms killed on exposure to the cement under fixed conditions. The germicidal efficiency of a dental cement is merely one of the properties which are of importance. Many other physical properties such as resistance to saliva, hardness, crushing strength, constancy of volume, etc., are also of importance.

**Ferric Chloride Solution, Determination of Free Acid in.** G. Romijn. (*Amer. J. Pharm.*, 1915, 87, 246.) The tests for oxychloride and for free acid in  $\text{Fe}_2\text{Cl}_6$  solution, as given in

various pharmacopœias, are not satisfactory. The following method is stated to be more satisfactory for the determination of free acid. It depends on the use of Fernbach and Wolf's soluble starch as a protecting colloid to prevent the precipitation of ferro-ferric hydrate. The following reagents are required : (a) Normal sodium thiosulphate solution containing 24.8 Gm. sodium thiosulphate in 100 c.c. (b) Cupric chloride starch. Mix 1 Gm. of  $\text{CuCl}_2$  with 49 Gm. of soluble starch previously dried at  $100^\circ\text{C}$ . The titration is effected as follows : To the cooled solution of 0.5 Gm.  $\text{CuCl}_2$  starch in 50 c.c. of water, contained in an Erlenmeyer flask of 100 c.c. capacity, add 2 c.c. of the ferric chloride solution to be tested. N/thiosulphate is now added, 5.5 c.c. for U.S.P.  $\text{Fe}_2\text{Cl}_6$  solution and 9 c.c. for that of the P.Ned. IV. The decolorized liquid is coloured fairly strong with methyl-orange solution. If the reaction of the mixture is acid, it is titrated drop by drop with N/10 NaOH. If the liquid is alkaline, it is rejected and the operation repeated after adding 1 c.c., or as much as may be required, of decinormal hydrochloric acid before adding the other reagents. As the result of a number of experiments it is concluded that the following limit for free acid may be stated : 2 c.c. of the solution should require no more than 1.2 c.c. of N/10 NaOH when examined in the above-mentioned manner. If the mixture be alkaline toward methyl-orange, the addition of 1 c.c. N/10 HCl to the  $\text{Fe}_2\text{Cl}_6$  solution should produce a mixture of acid reaction.

**Glass, Pharmaceutical, Ingredients for making.** (*Analyst*, 1915, 40, 264.) The Glass Research Committee of the Institute of Chemistry, among a number of formulæ for glass of different grades, give the following for a resistant glass suitable for pharmaceutical purposes, ampoules, etc. : Sand, 67.0 parts ; Alumina ( $\text{Al}_2\text{O}_3$ ), 10.0 ; Calcium carbonate, 12.5 ; Magnesia, 0.5 ; Potassium nitrate, 1.0 ; Sodium carbonate ( $\text{Na}_2\text{CO}_3$ ), 17.0 ; Boric anhydride ( $\text{B}_2\text{O}_3$ ), 8.0. This glass is intermediate in hardness between soft glass and combustion tubing, is highly resistant to chemical action, withstands changes of temperature well, and should be a very suitable glass for high-class beakers, flasks, etc.

**Glass for Ampoules and Medicine Bottles, Examination of.** L. Kroeber. (*Apoth. Ztg.*, 1914, 29, 974-8 ; *Chem. Abstr.*, 1915, 9, 1675.) Fiolax Jena glass answers the strict requirements for ampoules and especially stands the test of Mylius. The

scale of sensitiveness towards free alkali in decreasing order is narcotine hydrochloride, strychnine nitrate, phenolphthalein,  $\text{HgCl}_2$ , morphine hydrochloride. The German Ministry of War requires this test : Fill the rinsed ampoules with weak phenolphthalein solution, heat the tubes, close them by fusion, heat again to  $100^\circ\text{C}$ . for 30 minutes. The contents must not show a red colour. Anneler's test with 0.1 per cent. narcotine hydrochloride solution is by far the most sensitive. For medicinal glass in general, many possibilities of dangerous precipitates in inferior glass are cited. The author recommends filling the bottles with  $\text{CO}_2$ -free water containing 2-3 drops of phenolphthalein in 100 c.c. Heat in a current of steam for at least one hour. Allow to stand 24 hours. The contents should be colourless or faint pink, and the colour should disappear upon adding 2-3 drops of  $\text{N}/10$   $\text{HCl}$ . The following summary of treatment is given : Sterilize in a current of steam for  $\frac{1}{2}$ -1 hour. After 24 hours distilled  $\text{H}_2\text{O}$  should show no glittering silicates. Morphine-hydrochloride solution 1-2 per cent. should show at most a faint yellow colour. Strychnine nitrate 0.6 per cent. should not deposit crystalline needles.  $\text{HgCl}_2$  should not precipitate coloured oxides. Phenolphthalein, see above. Narcotine hydrochloride solution 0.1 per cent. at room temperature should not precipitate, or only very slightly, after 1 hour. Sterilize for 1 hour with a 1 per cent.  $\text{HCl}$  solution, then wash until litmus no longer shows acid. (See also *Y.B.*, 1914, 224, 228.)

**Glassware, Chemical, Composition of some Types of.** F. W. Branson. (*J.S.C.I.*, 1915, 34, 471.) The author has ascertained by analysis the composition of various types of chemical glass ware, more specially that used for beakers and flasks, and of miner's-lamp glass. For the former, a zinc borosilicate glass was found to be satisfactory and has been adopted. It answers all tests for stability. Experiments are being made with hard soda-glass, which works out well in the blowpipe flame. Analyses are given of Jena and Kavalier combustion tubing. A miner's-lamp glass, which is very tough and durable, has also been made successfully, based on the analysis of Jena miner's-lamp glass.

**Heavy Metals, Microchemical Detection of, by means of Caesium and Rubidium Salts.** M. Wagenaar. (*Pharm. Zentralh.*, 1914, 55, 699.) Metals of the Ag group, Ag, Hg, and Pb, give no reactions. As gives no reaction, but Sb reacts with both.

The formation of caesium-antimony iodide is evident with 0.01 micro-Gm. in a micro-c.c., and the same is true of the rubidium compound. Two separate droplets of the solution are treated, one with a minute particle of  $\text{RbCl}$ , the other with a similar particle of  $\text{KI}$ , and the two are mixed. A droplet of the  $\text{Sb}$  is then introduced. Handsome dark red doubly refractive crystals indicate the formation of the double salt. In this manner 2 micro-Gm. of  $\text{Sb}$  may be detected.  $\text{RbCl}$  is also a good micro-reagent for  $\text{Bi}$  salts, either alone or in conjunction with  $\text{KI}$ .  $\text{Cs}$  salts may also be used, and the presence of a small amount of  $\text{HNO}_3$  does not interfere with their reaction.  $\text{Pb}$  does not interfere with the  $\text{Cs-Sb}$  reaction; nor do  $\text{Cu}$ ,  $\text{Cd}$ , and  $\text{Hg}$ ; these metals only slightly affect the  $\text{Rb-Sb}$  reaction. One micro-Gm. of  $\text{Cu}$  may be detected by means of  $\text{RbCl}$  and 0.1 micro-Gm. with  $\text{CsCl}$ .  $\text{Cd}$  in  $\text{HCl}$  forms typical crystals with  $\text{RbCl}$  which will detect 0.01 micro-Gm. of  $\text{Cd}$ ;  $\text{CsCl}$  is rather less sensitive, it will detect 0.5 micro-Gm.  $\text{Co}$  does not react with  $\text{RbCl}$ ; with  $\text{CsCl}$  it gives fine blue prisms. The reaction is sensitive to 5 micro-Gm. of  $\text{Co}$ . Acids prevent it. These reagents are not suited for the detection of  $\text{Ni}$  or  $\text{Fe}$ .  $\text{Al}$  as  $\text{Al}_2\text{Cl}_6$  cannot be directly detected by  $\text{CsCl}$  or  $\text{RbCl}$ ; but if  $\text{KHSO}_4$  be added characteristic crystals are obtained. The same is the case with  $\text{Cr}$ . Two micro-Gm. of  $\text{Al}$  and 10 micro-Gm. of  $\text{Cr}$  can be thus detected.  $\text{Mn}$  gives no reaction.

**Hydrogen Dioxide, Delicate Reaction for.** *Rogai.* (*Giorn. farm. Chim.; Drugg. Circ.*, 1915, 59, 369.) When two or three drops of  $\text{KCNS}$  solution and five or six drops of  $\text{Et}_2\text{O}$  are added to two or three drops of a freshly prepared solution of  $\text{FeSO}_4$ , and this is followed by a small amount of the liquid under examination, the mixture will assume a pink or blood-red colour in the presence of  $\text{H}_2\text{O}_2$ , the intensity of colour varying with the quantity of the  $\text{H}_2\text{O}_2$  present. The author states that by this reaction as little as 0.0000144 Gm. of  $\text{H}_2\text{O}_2$  can be detected.

**Hydrogen Peroxide, Determination of Acidity of.** *T. Callan.* (*Pharm. J.*, 1915 [4], 40, 413.) The U.S.P. directs the acidity of  $\text{H}_2\text{O}_2$  solution to be determined by back titration. Five c.c. of  $\text{N}/10$   $\text{NaOH}$  is added to 25 c.c. of  $\text{H}_2\text{O}_2$ ; the free  $\text{NaOH}$  is then titrated with  $\text{N}/10$  acid with phenolphthalein indicator. In the B.P. the determination of free acid is made by direct titration with  $\text{N}/10$   $\text{NaOH}$  with methyl-orange indicator. The U.S.P. method gives results about 3 times as high as the B.P. method.

This is mainly due to the presence of phosphates in the commercial liquid, practically all of them containing these. In fact, both methods are correct, as far as they go. The U.S.P. method includes the phosphates and  $\text{CO}_2$  among the acids. The question arises, what is to be understood by the term "acidity"?

**Hydrogen Peroxide, Preservation of.** J. H. Walton, jun., and R. C. Judd. (*Z. physik. Chem.*, 1913, **83**, 315; *J.S.C.I.*, 1915, **33**, 1086.) Concentrated solutions of NaCl will preserve  $\text{H}_2\text{O}_2$  solutions; the time required for the decomposition of half the  $\text{H}_2\text{O}_2$  in a solution is approximately doubled by the addition of 1 mol. NaCl per litre.  $\text{H}_2\text{SO}_4$  even in a concentration of 0.00066 Gm. per litre has a pronounced retarding influence on the decomposition. A still better preservative is acetanilide, which is effective even in solutions containing positive catalytic agents, such as NaOH, and also soluble matter from glass.

**International Atomic Weights, 1915.** (*Analyst.*, 1915, **40**, 1.) O=16. Aluminium (Al), 27.1; Antimony (Sb), 120.2; Argon (A), 39.88; Arsenic (As), 74.96; Barium (Ba), 137.37; Bismuth (Bi), 208.0; Boron (B), 11.0; Bromine (Br), 79.72; Cadmium (Cd), 112.40; Cæsium (Cs), 132.81; Calcium (Ca), 40.07; Carbon (C), 12.00; Cerium (Ce), 140.25; Chlorine (Cl), 35.46; Chromium (Cr), 52.0; Cobalt (Co), 58.97; Columbium (Cb), 93.5; Copper (Cu), 63.57; Dysprosium (Dy), 162.5; Erbium (Er), 167.7; Europium (Eu), 152.0; Fluorine (F), 19.0; Gadolinium (Gd), 157.3; Gallium (Ga), 69.9; Germanium (Ge), 72.5; Glucinum (Gl), 9.1; Gold (Au), 197.2; Helium (He), 3.99; Holmium (Ho), 163.5; Hydrogen (H), 1.008; Indium (In), 114.8; Iodine (I), 126.92; Iridium (Ir), 193.1; Iron (Fe), 55.84; Krypton (Kr), 82.92; Lanthanum (La), 139.0; Lead (Pb), 207.10; Lithium (Li), 6.94; Lutecium (Lu), 174.0; Magnesium (Mg), 24.32; Manganese (Mn), 54.93; Mercury (Hg), 200.6; Molybdenum (Mo), 96.0; Neodymium (Nd), 144.3; Neon (Ne), 20.2; Nickel (Ni), 58.68; Niton (radium emanation) (Nt), 222.4; Nitrogen (N), 14.01; Osmium (Os), 190.9; Oxygen (O), 16.00; Palladium (Pd), 106.7; Phosphorus (P), 31.04; Platinum (Pt), 195.2; Potassium (K), 39.10; Praseodymium (Pr), 140.6; Radium (Ra), 226.4; Rhodium (Rh), 102.9; Rubidium (Rb), 85.45; Ruthenium (Ru), 101.7; Samarium (Sa), 150.4; Scandium (Sc), 44.1; Selenium (Se), 79.2; Silicon (Si), 28.3; Silver (Ag), 107.88; Sodium (Na), 23.00; Strontium (Sr), 87.63; Sulphur (S), 32.07; Tantalum (Ta), 181.5; Tellurium (Te), 127.5;



Terbium (Tb), 159.2 ; Thallium (Tl), 204.0 ; Thorium (Th), 232.4 ; Thulium (Tm), 168.5 ; Tin (Sn), 119.0 ; Titanium (Ti), 48.1 ; Tungsten (W), 184.0 ; Uranium (U), 238.5 ; Vanadium (V), 51.0 ; Xenon (Xe), 130.2 ; Ytterbium (Neoytterbium) (Yb), 172.0 ; Yttrium (Yt), 89.0 ; Zinc (Zn), 65.37 ; Zirconium (Zr), 90.6.

**Magnesium Carbonate, Absorptive Power of, towards Volatile Substances.** J. M a r a n n e. (*L'Union Pharm.*, 1914, 55, 517.) A block of compressed hydrated  $\text{MgCO}_3$  kept near camphor, but separated therefrom by three layers of stout paper for about two months, was found to have but a slight camphoraceous odour. When it was triturated with water, the odour was more pronounced. When the carbonate was dissolved in citric acid solution, the camphor odour became very marked, as pronounced almost as that of camphorated spirit. This points to the necessity of storing  $\text{MgCO}_3$  [and similar powders] away from strongly odorous substances, to prevent taint.

**Magnesium Citrate, Official, of the French Codex.** E. L é g e r. (*J. Pharm. Chim.*, 1915, 11, 157.) Although apparently simple, the preparation of magnesium citrate which will have the requisite solubility in water of 1 : 2 requires special manipulation. The method of the French Codex is not satisfactory, consequently commercial samples of magnesium citrate are not uniform in properties and solubility. The author modifies the official process thus : Citric acid, 100 Gm., is dissolved with gentle heat in water 35 Gm., and the solution is cooled. Hydrated magnesium carbonate, 35 Gm., is sifted and placed in a capacious dish. The acid solution is added to it, and intimately mixed by means of a wooden spatula until it gradually becomes a homogeneous pasty, spongy mass. It is then put on in pieces the size of a walnut on sheets of glass and dried between  $40^\circ$  and  $50^\circ\text{C}$ . In 2 or 3 hours, these light spongy pieces will be hard outside, the inside being soft and translucent ; but becoming hard when cold. They are then powdered and dried by exposure to the air for two or three days. The product is the magnesium citrate  $\text{Mg}_3\text{2C}_6\text{H}_5\text{O}_7 + 13\text{H}_2\text{O}$  ; soluble in hot water 1 : 2. The solubility test should be thus expressed : One Gm. of the citrate should dissolve with slight effervescence in 2 c.c. of water at  $70^\circ\text{C}$ ., when the containing vessel is kept in a water-bath at  $70^\circ\text{C}$ . The official statement that the salt contains 4 mols.  $\text{H}_2\text{O}$  is a misprint for 14 mols., the figure originally found by Heldt. The

latter however is erroneous. The author finds the correct degree of hydration is equivalent to 13 mols. One Gm. of the above citrate gives from 0.16 to 0.17 Gm. of MgO when ignited.

**Mercuric Iodide, Determination of, in Tablets.** A. W. Bender. (*J. Ind. Eng. Chem.*, 1914, **6**, 753.) Powder a sufficient number of tablets to represent 1 to 2 grains of mercuric iodide. Place in a 180 c.c. Erlenmeyer flask and add 20 c.c. of 1:1 HCl. Add about 0.5 Gm.  $\text{KClO}_3$  and stopper with a glass tube reflux condenser. Digest on the sand-bath until the mercuric iodide is all dissolved. Cool and dilute with water to about 100 c.c. Wash glass tube condenser and remove. Blow out chlorine with a current of air. Filter and wash insoluble matter by decantation. Make filtrate alkaline with ammonia and precipitate immediately in the cold with a slow stream of  $\text{H}_2\text{S}$ . Let stand for a few hours and filter through a weighed Gooch crucible. Wash with water and alcohol, dry at  $100^\circ\text{C}$ . and weigh. Grams of  $\text{HgS} \times 30.17 = \text{grains of HgI}_2$ . Grams of  $\text{HgS} \times 1.955 = \text{grams of HgI}_2$ .

**Metals, Methods for Reducing, in Crystalline Form on Micro-slides.** J. H. Bowman. (*J. Amer. Chem. Soc.*, 1915, **37**, 1468.) Simple methods are given for preparing Au, Cu, Pb, Bi, Sn and Cd in small crystals and for mounting them as permanent micro-objects. Microphotographs of these are reproduced.

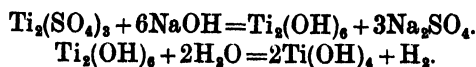
**Nessler's Reagent, Preparation of.** Frerichs and Mannheim. (*Apoth. Zeit.; Drugg. Circ.*, 1915, **59**, 167.)  $\text{HgI}_2$  3.5 Gm. is dissolved in a solution of KI 2.5 Gm.; in water 3 c.c. A solution of KOH 15 Gm. in water to 100 c.c. is added. After standing for several days the clear liquid is decanted, or filtered through a sand filter. For  $\text{NH}_3$  in water from 1 to 2 c.c. of the reagent should be used for 10 c.c. of the water to be tested. For  $\text{NH}_3$  in hexamine, 10 c.c. of the aqueous solution, 1:20, is mixed with 20 drops of the reagent. For detecting aldehydes in  $\text{Et}_2\text{O}$ , 10 c.c. of the latter is treated with 1 c.c. of the Nessler's solution. The reagent prepared according to the above formula is claimed to be more reliable than that made by other methods. (See also *Y.B.*, 1910, 135, and *Gen. Index*.)

**Nickel,  $\alpha$ -Benzildioxime to Detect Traces of.** — Attack. (*Chem. Zeit.*, 1913, 773; *Pharm. Zentralh.*, 1914, **55**, 700.) Bessides being much cheaper than dimethylglyoxime, which has

been used as a Ni-precipitant,  $\alpha$ -benzildioxime is at least as efficient. It is prepared by dissolving benzil 10 Gm. in MeOH 50 c.c., and boiling for 8 hours with a strong aqueous solution of hydroxylamine hydrochloride 8 Gm. The precipitated dioxime is washed with water and EtOH and crystallized from acetone. An EtOH solution of this  $\alpha$ -benzildioxime gives a red precipitate with Ni compounds insoluble in water, EtOH, acetone and AmOH. An evident reddish precipitate is obtained with a dilution of Ni of 1 : 400,000. The precipitation is quantitative and may be applied to the gravimetric determination of Ni. The empirical formula of the compound is  $C_{28}H_{22}N_2O_4Ni$ , an atom of Ni combining with 2 mols. of the dioxime.

**Nitrates, New Qualitative Colour Reaction for.** A. Tingle. (*J.S.C.I.*, 1915, 34, 393.) Two Gm. of salicylic acid is dissolved in 30 c.c. of  $H_2SO_4$  (sp.g. 1.84). The test is applied as follows : If the substance to be tested is a solid, it is cautiously heated in a test tube with a slight excess of the reagent. A drop of the resulting liquid is withdrawn on to a cold white porcelain slab and two or more drops of concentrated KOH solution are added—enough to make the drop alkaline. The development of a yellow or orange colour indicates the presence of nitrate. If the substance to be tested is in solution, two or three drops are placed in an evaporating dish, two or three drops of the reagent are added and the mixture is warmed cautiously over a Bunsen flame till acid fumes are evolved. When cool, a slight excess of KOH is added, as directed above. A distinct yellow colour is given by one drop of a 0.1 per cent. solution of potassium nitrate. Strong AmOH may be substituted for KOH solution, but the yellow colour produced is more fugitive. The presence of halides does not interfere with this test.

**Nitrates, Rapid Method for Determining.** E. Knecht. (*J. Soc. Chem. Ind.*, 1915, 34, 126.) The method depends on the fact that titanous hydroxide is capable of effecting the complete reduction of nitrates (and nitrites) to ammonia. When NaOH is added to a solution of a titanous salt, black titanous hydroxide is precipitated, and this begins to decompose almost at once, yielding nascent H and the white titanic hydroxide, probably according to the following equations :



A convenient amount of nitrate for a determination is about the equivalent of 0.1 Gm. of  $\text{KNO}_3$ . Ten c.c. of a 1 per cent. solution are measured into a Cu flask, excess of  $\text{NaOH}$  is added, and then about 20 c.c. of commercial titanous sulphate or chloride. The solution is at once distilled into standard acid, and the liberated  $\text{NH}_3$  estimated. The reagents must be added in the order stated, or there will be a loss of nitrous or nitric oxide.

**Plaster of Paris, for Surgical Casts.** E. Canals. (*J. Pharm. Chim.*, 1915, 11, 118, 286.) In almost all the plasters of Paris examined  $\text{MgSO}_4$  and  $\text{Na}_2\text{SO}_4$  were present in equal proportions, and sometimes in notable quantity. The more impure plasters contained the larger amounts of these two salts. They have an important influence on the time of setting of the moist magma. When present in only a few tenths per cent., they lessen by 6 minutes. When the dry powder contains a few percentages the period of setting of the plaster is reduced to one-third the normal time.  $\text{Na}_2\text{SO}_4$  is more active in this respect than  $\text{MgSO}_4$ . They may be recommended as an admixture in surgical plaster of Paris as agents for hastening the setting, since they do not occasion undue rise in temperature when the mass is made with water. This obviates the danger of scalding such as has been known to occur when  $\text{NaCl}$  is used for the purpose.

**Pt, Detection of, with  $\text{SnCl}_2$ .** E. Langstein and P. H. Prausnitz. (*Chem. Ztg.*, 38, 802; *Chem. Abstr.*, 1914, 8, 3401.) Wöhler has described a test for Pt in  $\text{HCl}$  solution with  $\text{SnCl}_2$  by which a blood red (in weaker solutions a golden brown) colour is produced. By shaking with  $\text{Et}_2\text{O}$  all the colour passes into the latter, the absorption spectrum of the  $\text{Et}_2\text{O}$  solution showing a characteristic band between 750 and 533  $\mu$ . Although Ir, Pd, Au and small amounts of Fe do not interfere, all organic matter (e.g. filter paper or humus), even after treatment with aqua regia, gives a brown colour which is partly extracted by  $\text{Et}_2\text{O}$  to which it gives the same colour Pt. Moreover, the absorption spectrum also is the same as for Pt. In consequence before extracting ores with aqua regia, it is advisable to fuse the sample with  $\text{K}_2\text{S}_2\text{O}_7$ .

**Platinum Residues, Working up.** D. J. De Jong. (*Chem*

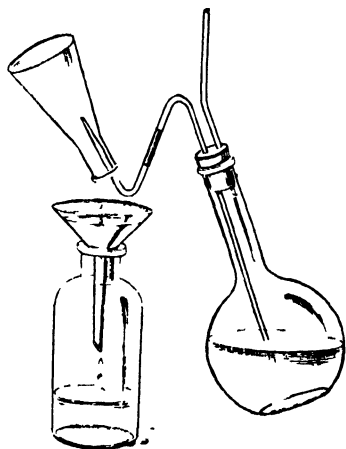
*Weekblad*, 10, 833; *Chem. Abst.*, 1914, 8, 635.) Place the residues in a large porcelain bowl (2 litres capacity for 100 Gm. Pt), evaporate off most of the liquid, make alkaline with NaOH, and while stirring add 50 Gm. NaCOOH little by little. If the liquid foams add a little more NaOH. Heat for 1 hour with occasional stirring on the water-bath, then with stirring add 25 per cent. HCl slowly until acid, and heat for an hour longer. Filter off the Pt with suction on a soft paper, wash twice by decantation with hot 2 per cent. HCl, then with hot water till free from acid, separate the Pt from the filter (which should not be added), dry, ignite and weigh. Pour over the Pt 5 times its weight of 25 per cent. HCl, heat on the water-bath, and add little by little 50 per cent. HNO<sub>3</sub> until no more gas is evolved (about 1 c.c. HNO<sub>3</sub> per Gm. Pt). Evaporate in a porcelain bowl 3 times to small bulk to get rid of HNO<sub>3</sub>, then 2 or 3 times with water to get rid of the bulk of the HCl, dilute with an equal bulk of water, filter after cooling on a soft counterpoised filter using suction (filter again if necessary through a double filter to catch the last traces of C), wash, dry and weigh the filter + residue, deduct from the weight of the Pt, and then make the Pt solution up to the strength desired. This gives a Pt solution free from K.

**Poppy Capsules, Magnesium Phosphate in.** J. E. Kimbrell. (*J. Amer. Pharm. Assoc.*, 1915, 4, 623.) MgHPO<sub>4</sub> occurs in poppy capsules. Its presence may possibly vitiate morphinometric assays of the drug and its preparations when the morphine is crystallized out from ether in presence of AmOH. Under these conditions crystals of MgNH<sub>4</sub>PO<sub>4</sub> are likely to form simultaneously, and might be weighed with the morphine.

**Potassium Carbonate, KClO<sub>3</sub> as Impurity in.** W. Kohen. (*Chem. Zeit.*, 1914, 38.) Samples of K<sub>2</sub>CO<sub>3</sub>, which gave only a trace of AgCl when dissolved in excess of HNO<sub>3</sub> and tested with AgNO<sub>3</sub>, were found to give a considerable reaction if the test was applied after heating to redness. That this was due to KClO<sub>3</sub> was proved by the reaction obtained with KI and starch paste with a solution of the K<sub>2</sub>CO<sub>3</sub> in excess of HCl. The samples of K<sub>2</sub>CO<sub>3</sub> containing this impurity were of electrolytic origin.

**Precipitates, Insoluble, Transference of, to Tared Filters.** M. François and E. Lasausse. (*J. Pharm. Chim.*, 1915,

11, 233.) The illustration sufficiently explains the modification of the jet of the wash bottle which renders the washing out of adherent precipitates easy. The conical form of precipitating flask is much to be preferred. Very adherent precipitates may first be moved and broken up with a rubber-tipped rod.



**Radium and Mesothorium Preparations of Unequal Age, Distinguishing between, by Means of their Radiation.**

Otto Hahn. (*Le radium*, 11, 71; *Chem. Abstr.*, 1914, 8, 3268.) With a view of finding some method of distinguishing between Ra pre-

parations and Meso-Th preparations of unequal age a study was made of the absorption of the  $\gamma$ -rays for 5 such preparations chosen as follows: (1) pure  $\text{RaBr}_2$ ; (2) freshly prepared commercial (Meso-Th)- $\text{Br}_2$ ; (3) (Meso-Th)- $\text{Br}_2$  two years old; (4) new (Meso-Th) $\text{Br}_2$  free from Ra; and (5) Radio-Th separated from Meso-Th a long time ago and purified by precipitation. An examination of the absorption curves of the  $\gamma$ -radiation in Pb, determined with the same electroscope of Pb 3.3 mm. in thickness, showed that it was possible to distinguish closely between all the preparations. If the radiation passing through 3.3 mm. be taken as 100 for each of the preparations, then the radiation unabsorbed on passing through 45 mm. of Pb for each are, respectively, 9.0, 7.5, 9.1, 6.8 and 12.4. The Ra and the old Meso-Th have thus initially and finally the same ratio, but they may be distinguished with intermediate thicknesses, as between 10 and 20 mm.

**Rat Pastes, Phosphorus Tablets, Phosphorated Resin and Similar Preparations, Determination of P in.** G. E. E'We and C. E. Vanderkleed. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1683.) The following method works well with phosphorus rat pastes: Take a sample of about 1 Gm., place in a distilling flask connected with a  $\text{CO}_2$  generator and a condenser, and connect the condenser with a 300 c.c. Erlen-

meyer flask containing 50 c.c. of 3 per cent.  $\text{AgNO}_3$  solution. Connect the flask with a series of two U tubes containing 3 per cent.  $\text{AgNO}_3$  solution. All connections exposed to the phosphorus must be of glass or of cork covered with plaster of Paris. Pass  $\text{CO}_2$  through the apparatus for 20 minutes, testing for leaks with flexible collodion, which will bubble at a leak. Place 125 c.c. of cold, freshly boiled, distilled water containing 2 c.c. of  $\text{H}_2\text{SO}_4$  in the flask containing the sample by means of the tube which leads to the  $\text{CO}_2$  generator. Continue to pass in  $\text{CO}_2$ , and heat the flask gently until, after about 3 hours, practically all of the liquid in the flask has been distilled into the  $\text{AgNO}_3$  solution. Finally, allow the condenser to become hot from the distillation, and disconnect between generator and distillation flask before removing flame. Collect all the  $\text{AgNO}_3$  solutions in the Erlenmeyer flask, using  $\text{HNO}_3$  to dissolve the black precipitate in the U tubes. Add 15 c.c. of  $\text{HNO}_3$  to the mixture, boil for 5 minutes, and add  $\text{HCl}$ , in moderate excess, to precipitate the silver. Boil for 20 minutes, let cool, filter, and concentrate to 150 c.c. Cool to  $40^\circ\text{C}$ ., add 100 c.c. ammonium molybdate solution, stir well, and let stand in warm place over night. Filter off clear liquid, wash precipitate by decantation, using 25 c.c. water for each washing, transfer to filter, and wash until two fillings of the funnel are rendered pink by one drop of  $\text{N}/2$   $\text{KOH}$  solution, using phenolphthalein as indicator. Place the filter and precipitate in a glass-stoppered flask, add an excess of  $\text{N}/2$   $\text{KOH}$  solution, shake for a few minutes, add phenolphthalein indicator, and titrate back with  $\text{N}/2$   $\text{H}_2\text{SO}_4$ . Each c.c. of  $\text{N}/2$  potassium hydroxide solution is equivalent to 0.0007071 Gm. phosphorus.

**Selenium and Tellurium Acids and Salts, New Test for.** G. Denigès. (*Annales Chim. analyt.*, 1915, 20, 57.) The author's  $\text{HgNO}_3$  reagent ( $\text{HgNO}_3$  10 ;  $\text{HNO}_3$  sp.g. 1.39, 10 ; water 100) is useful to precipitate the free acids of Se or their salts. With *selenic acid* or its salts, on adding half a volume of the reagent to a volume of a solution of 1 : 1000 or stronger, a white creamy precipitate is formed which soon develops a microcrystalline character the shape of the crystals as seen *sub lente* being modified by the strength of the solution. With *selenious acid* and its salts the reaction is still more sensitive for mercurous selenite is very insoluble, and from strong solutions the precipitate is crystalline in aggregated micro needles.

The reaction may be obtained with ease with drops on a microslide. By special manipulation, described in detail, the reaction may be used to identify very minute quantities of selenium by the microscope. With *telluric acid* or a *tellurate*, a yellow precipitate of mercurous tellurate is formed which also rapidly becomes crystalline. The micro appearance of all three crystalline forms is shown.

**Silver, Estimation of, in Argentum proteinicum and Similar Preparations.** Stoecker. (*Apoth. Ztg.*, 29, 344-5.) Dissolve 1 Gm. of the sample in 10 c.c. of distilled water, add 10 c.c.  $\text{HNO}_3$  (Ph. G. IV.), mix and add 10 c.c. 10 per cent.  $\text{NaNO}_2$  solution, then heat the mixture until  $\text{HNO}_3$  is expelled, finally adding a few drops  $\text{EtOH}$  to eliminate any frothy coating. Titrate with N/10 AmCNS and iron alum indicator.

**Sulphur, Analysis of.** M. G. Levi. (*Annali Chim. Appl.*, 1915, 1, 9; *J.S.C.I.*, 1915, 34, 282.) About 0.2 Gm. of the S is weighed directly into a 100 c.c. conical flask, which is then fitted, by means of a ground glass joint, with a reflux tube. The flask is immersed in cold water, and whilst held in an inclined position 10 c.c. of fuming  $\text{HNO}_3$  (sp.g. 1.52) and 5 drops of Br are introduced successively through the reflux tube. The flask is shaken occasionally until most of the S and Br is dissolved, whereupon a further 5 c.c. of  $\text{HNO}_3$  is added, and after heating for half an hour on the water-bath, the flask is again immersed in cold water, the solution diluted with 50 c.c. of water added in small portions through the reflux tube, and the S determined as  $\text{BaSO}_4$  in the usual way. With low-grade sulphur, the determination should always be made with the S separated from the original sample by extraction with  $\text{CS}_2$ .

**Sulphuric Acid, Determination of As, Fe and Hg in.** H. Nisenson. (*Chem. Ztg.*, 1914, 38, 1097; *Chem. Abstr.*, 1915, 9, 278.) Heat 100 c.c. of the sample on a sand-bath until no more nitrous fumes appear. Dilute with 3 vols. of  $\text{H}_2\text{O}$ . In a large Erlenmeyer with a 2-hole stopper, place 60 Gm. of Fe-free Zn. Through one of the holes in the stopper run a funnel with a tube reaching to the bottom of the flask. Through the other hole run a glass tube leading into a small Erlenmeyer flask which contains 100 c.c. of  $\text{H}_2\text{O}$ , 1 c.c. of Br and 1 drop of  $\text{H}_2\text{SO}_4$ . The sample, after heating, diluting and cooling, is poured through the funnel, care being taken



that a violent evolution of H does not occur. When all the acid has been added, nearly all of the Zn should be in solution. Wash the funnel with a little dilute  $\text{H}_2\text{SO}_4$ . The undissolved spongy metal is filtered off and washed. Determine the Fe in the filtrate by titration with N/100  $\text{KMnO}_4$ . Dry the metal on the filter by warming gently, mix it with freshly ignited  $\text{CaO}$  in a porcelain crucible and cover the crucible with a gold cover. Place  $\text{H}_2\text{O}$  in the depression in the upper side of the cover and apply a gentle heat. After heating for 10 minutes, wash the cover with  $\text{EtOH}$  and dry. Determine the Hg by igniting the cover and noting the loss. The As which has been converted to  $\text{AsH}_3$  and absorbed by the Br is determined as follows : Drive off the excess of Br by heating on a sand-bath, reduce with  $\text{Na}_2\text{SO}_3$  and titrate the  $\text{As}_2\text{O}_3$  with N/10  $\text{KBrO}_3$  solution.

**Sulphurous Acid, Micro-detection of, in the Atmosphere.** G. Denigès. (*Bull. Soc. Pharm. Bordeaux ; Annales Chim. analyt.*, 1915, 20, 10.) The formation of characteristic micro-deposits is the basis of the method. Three mercurial reagents are employed. *Mercuric sulphate* :  $\text{HgO}$ , 5 Gm., is dissolved in  $\text{H}_2\text{SO}_4$ , sp.g. 1.84, 20 c.c., and the solution is made up to 100 c.c. with distilled water. A glass stirrer with a rounded end is dipped in this, then introduced into the air to be tested. In presence of  $\text{SO}_2$ , the moistened end becomes coated with a white crystalline deposit, which when examined by the microscope show fernleaf growths resembling those of  $\text{MgNH}_4\text{PO}_4$ , illustrations of which are given. *Mercuric acetate* : The reagent is prepared with  $\text{HgO}$ , 5 Gm. ; glacial  $\text{HC}_2\text{H}_3\text{O}_2$ , 1 Gm., made up to 100 c.c. with water. The method of testing is the same as above. Characteristic micro-sphaerocrystals, generally radiated, which do not attain their full size for 5 minutes. If left for some time elongated pointed crystals are also formed. Figures of these are given. *Mercurous nitrate* :  $\text{HgNO}_3$ , 5 Gm., is dissolved in  $\text{HNO}_3$ , sp.g. 1.39, 5 c.c. and water, 50 c.c. In this case, when the atmosphere is tested in the same manner, a brownish coating is formed in the presence of  $\text{SO}_2$  due to the presence of reduced Hg and  $\text{Hg}_2\text{SO}_4$ . The structure is rarely crystalline.

**Water Analysis, Rapid Determination of Mg by Titration in the Presence of Calcium.** V. Froboese. (*Z. anorg. Chem.*, 1914, 89, 370-9 ; *Chem. Abstr.*, 1915, 9, 1644.) Ca is precipitated as  $\text{CaC}_2\text{O}_4$ , and Mg is titrated with potassium palmitate

solution, without removing the precipitate. Ca is determined by difference. Two hundred c.c. (samples up to 1 litre may be used) of the water are heated to boiling; 1 drop methyl orange is added; then an excess concentrated  $\text{H}_2\text{C}_2\text{O}_4$  solution; then enough 50 per cent. KOH just to give the yellow colour (an excess is to be avoided). The solution on cooling should regain a slightly reddish tint. To the cooled solution add 5 drops phenolphthalein and just enough N/10 KOH to give a pale rose tint, then titrate with N/10 potassium palmitate. In the Winkler method instead of taking the end point as the rather indefinite "permanent lather," the author titrates until the sound of the bubbles breaking can no longer be detected when holding the titrating flask close to the ear.

**Water, Drinking, Bacteriological Standard for.** J. F. A n d e r s o n. (*U.S. Public Health Reports*, 1914, **29**, 2959-66; *Chem. Abstr. Amer. Chem. Soc.*, 1915, **9**, 676.) The bacteriological standard adopted by the U.S. Treasury Department for drinking water supplied to the public is as follows: (1) The total number of bacteria developing on standard agar plates, incubated 24 hours at  $37^\circ\text{C}$ ., shall not exceed 100 per c.c. Provided, that the estimate shall be made from not less than 2 plates, showing such numbers and distribution of colonies as to indicate that the estimate is reliable and accurate. (2) Not more than one out of five 10 c.c. portions of any sample examined shall show the presence of organisms of the *B. coli* group when tested according to special directions.

**Water, Object and Limitations of Bacteriologic Examination of.** W. H. F r o s t. (*Eng. Contr.*, **42**, 250; *Chem. Abstr. Amer. Chem. Soc.*, 1914, **8**, 3606.) A report on public water supplies drawn from the Ohio river at Wheeling, W. Va. Practical water examinations aim to determine the numbers of bacteria belonging to three general groups. (1) Bacteria developing on standard gelatin plates at  $20^\circ\text{C}$ . in 48 hours. This group includes a large proportion of harmless varieties. In general, water supplies taken from rivers and efficiently purified by filtration should not show more than 100 bacteria per c.c. on this medium. (2) Bacteria developing on standard agar at  $37^\circ\text{C}$ . This group includes a larger proportion of the varieties which normally live in the animal body and whose presence in water indicates probable pollution with discharges from the animal body. Really good water supplies will ordinarily show

not more than 10-50 bacteria per c.c. (3) The bacteria of the *B. coli* group should be absent in 10 c.c. of the water in 70-90 per cent. of the samples and should always be absent in 1 c.c. of the water.

**Water, Schardinger-Dunham Medium for Testing for the Presence of Putrefactive  $H_2S$  forming Bacteria in.** E. M. Chamo<sup>t</sup> and H. W. Redfield. (*J. Amer. Chem. Soc.*, 1915, 37, 1606.) In the course of an investigation of the different culture media employed in the bacteriological examination of water, the authors have first dealt with the peptone medium originally proposed by Schardinger, who detected the presence of  $H_2S$  by means of a  $PbCO_3$  test paper suspended over the culture. For the detection of "putrefactive" bacteria Dunham made a slight modification in the Schardinger medium: To about 90 c.c. of water, 10 c.c. of a 10 per cent. peptone, 5 per cent. salt solution, previously sterilized, were added. This gave a resulting solution containing 1 per cent. of peptone and 0.5 per cent. of NaCl. The mixture was made in a sterile Erlenmeyer flask, provided with a cotton plug. A strip of paper, impregnated with  $PbCO_3$ , was suspended over the mixture and the flask was then placed in the incubator at 37°C. for 24 hours. Under these conditions of temperature and nutrition, Dunham claimed that the colon bacillus and the bacteria of putrefaction readily multiply and the latter cause the production of hydrogen sulphide which discolours the lead paper. Although a few water analysts appear to have made use of the Schardinger-Dunham method in the examination of suspected waters, there is no published indication that the value of the test is appreciated, which the authors consider to be most useful. The chief drawback is that the indications are not obtained in a short enough time. With badly contaminated waters strong tests for  $H_2S$  can be obtained in from 24 to 28 hours, but with other waters from 3 to 4 days of incubation were necessary. Moreover, it appeared probable that the hydrogen sulphide rapidly formed was not due to the colon group but to some other class of bacteria, while the slowly evolved hydrogen sulphide might possibly be ascribed to colon group organisms rather than those classed by Dunham as putrefactive.

The authors have succeeded in improving the method, the experiments being fully described and summarized as follows: Irrespective of the inorganic salts present and of the acidity

of the medium, a concentration of between 3 and 4 per cent. of peptone in the final inoculated and incubated medium appears to be best for the most rapid and energetic production of  $H_2S$ . The addition of beef broth to simple peptone media slightly increases its sensitiveness, but not in proportion to the increased trouble and labour involved. If NaCl is used, the quantity added must not be over 1.5 per cent. Cultures to which this salt was added showed greater  $H_2S$  production than those which contained none. In 3 per cent. peptone media, the presence of from 0.5 per cent. to 1 per cent. of KCl had a decidedly beneficial influence and led to quicker, better and far more uniform results than any other inorganic salt tried. Positive results of  $H_2S$  formation may be obtained in 18 hours. No  $H_2S$  formation is obtainable in as long a period as 72 hours from natural waters which are truly "clean," while much is formed in from 12 to 24 hours with contaminated waters. The fæces of domestic animals contain bacteria which are capable of producing  $H_2S$  from a simple peptone medium in as large amounts as is the case of the bacteria from human fæces. The large amounts of  $H_2S$  rapidly produced by organisms of sewage appears to be not due primarily to members of the *B. coli* group. This group of  $H_2S$  producing bacteria do not actively ferment carbohydrates. Hence testing for their presence is a valuable aid supplementing tests for gas producers and is of especial value in polluted waters in which the *B. coli* group is absent. Some evidence has been obtained which apparently indicates that  $H_2S$  is more rapidly produced in waters containing a mixed bacterial flora than by the isolated pure cultures alone.

**Water, Sterilization of, for Drinking Purposes, with Chlorinated Lime Tablets.** H. Vincent and — Gaillard. (*Comptes rend.*, 1915, 160, 483.) Compressed tablets, each containing 0.015 Gm. of chlorinated lime and 0.08 Gm. of pure NaCl, are recommended for rendering even grossly polluted water fit for drinking after 15 to 20 minutes. One such tablet is sufficient for a litre of water, which, after such treatment, is practically devoid of taste.

**Waters, Aerated, Determination of Copper and Lead in.** C. Reese and J. Drost. (*Zeit. Untersuch. Nahr. Genussm.*, 1914, 28, 427; *Chem. Abstr.*, 1915, 9, 1517.) Cu and Pb were precipitated as sulphides, the CuS and PbS separated and the

amounts of each determined colorimetrically with the tint produced in standard solutions of each metal, as sulphides. The result was checked in the case of Pb by comparing the resultant opalescence on adding  $K_2CrO_4$  to definite volumes of the water and standard dilutions. The Cu by comparing its  $Cu_2FeCy_6$  tint with a standard. Zn when present was also detected by precipitating with  $K_4FeCy_6$ . By these methods as little as 0.01 Mgm. of Pb or Cu per litre can be detected and determined accurately to 0.10 Mgm. The waters produced in Germany from 100 different machines have been examined; the charge being left in the pressure chamber for 12 hours under the maximum pressure. Twenty-one machines delivered water with more than 0.35 Mgm. Pb per litre, 13 with more than 0.6 Mgm. and 9 with more than 1 Mgm. per litre. Cu exceeded 0.35 Mgm. per litre in water from 48 per cent. of the machines, over 0.6 Mgm. in 21 and over 1 Mgm. in 9. Zn was found only in minute traces.

**Zinc Peroxide.** Riesenfeld and Nottebohm. (*Zeit. anorgan. Chem.: Drugg. Circ.*, 1915, **59**, 167.) Zinc peroxide, which hitherto has not been known in a pure state, may be prepared by dissolving  $Zn_2NO_3$  in strong  $AmOH$  and adding to the solution, previously cooled to  $-5^\circ$ , concentrated  $H_2O_2$  solution. The mixture is allowed to stand for several hours, the liquid decanted and the precipitate collected on a filter, washed with water,  $EtOH$  and  $Et_2O$  and dried over  $CaCl_2$  and  $NaOH$ .  $ZnO_2$  thus prepared occurs as a white amorphous powder and has the formula  $(ZnO_2)_2H_2O$ . (See also *Y.B.*, 1910, 140.)

**Zinc-sodium Cyanide.** N. Herz. (*J. Amer. Chem. Soc.*, 1914, **36**, 45.) This salt,  $Na_2Zn(CN)_4 \cdot 3H_2O$ , is a powerful antiseptic, extremely soluble in water, forms orthorhombic prisms which rapidly effloresce in the air, forming a chalky, stable anhydrous compound; it is slowly but completely soluble in water. It is prepared by dissolving  $ZnCl_2$  in boiling water by means of a little free  $HCl$ , and adding a solution of  $NaCN$  previously boiled with  $CaO$  to remove any  $Na_2CO_3$ . The precipitated  $Zn(CN)_2$  is washed with cold recently boiled distilled water and digested in a solution of  $NaCN$  free from  $Na_2CO_3$ . After filtering to remove excess of  $Zn(CN)_2$ , the filtrate is evaporated to a syrupy consistence and crystallized over  $H_2SO_4$ .

## ORGANIC CHEMISTRY, UNCLASSIFIED

**Acetanilide and Phenacetin, Determination of, in Admixture.**  
W. O. Emery. (*J. Ind. and Eng. Chem.*, 1914, 6, 665.) The method proposed depends on the fact that, when phenacetin is added to a solution of I in KI containing a mineral acid, an iodine addition product or periodide separates out in a crystalline condition, whilst the corresponding product of acetanilide, if formed at all, remains soluble. A quantity of 0.2 Gm. of the phenacetin-acetanilide mixture is dissolved by warming with 2 c.c. of glacial acetic acid and 40 c.c. of water, and the solution is poured into a 100 c.c. flask containing 25 c.c. of N/5 I solution warmed previously to 40°C. Three c.c. of strong HCl acid is then added, the closed flask shaken gently until a crystalline precipitate appears, and the mixture allowed to cool. After dilution with water to about 97 c.c., the mixture is placed aside overnight, then diluted to 100 c.c., filtered, and 50 c.c. of the filtrate are titrated with thiosulphate solution. Each c.c. of N/10 I solution is equivalent to 0.00889 Gm. of phenacetin. The latter may also be estimated gravimetrically by collecting all the iodine precipitate on the filter, washing it with iodine solution, transferring it to a separating funnel and extracting the phenacetin with  $\text{CHCl}_3$ , after the free and combined iodine has been destroyed by the addition of  $\text{Na}_2\text{SO}_3$ . The acetanilide is estimated in the filtrate from the iodine precipitate; an aliquot portion of the filtrate is treated with  $\text{Na}_2\text{SO}_3$ , a slight excess of  $\text{NaHCO}_3$  is added, then 2 drops of acetic anhydride, and the solution is extracted with  $\text{CHCl}_3$ . The  $\text{CHCl}_3$  extract is filtered, evaporated to 20 c.c., 10 c.c. of dilute  $\text{H}_2\text{SO}_4$  is added, the mixture heated on a water-bath until reduced to half its volume, and, after a further heating for 1 hour with 20 c.c. of water and 10 c.c. of strong HCl, is titrated with KBr-KBrO<sub>3</sub> solution; each c.c. of the latter should be equivalent to 0.005-0.01 Gm. of acetanilide. When the phenacetin and acetanilide are mixed with caffeine and antipyrine, the mixture of the four substances is digested with dilute  $\text{H}_2\text{SO}_4$ , in order to convert the phenacetin and acetanilide into phenetidine and aniline sulphates, respectively, from which caffeine and antipyrine may be separated by shaking out with  $\text{CHCl}_3$ . The phenacetin and acetanilide are then regenerated by treating the acid aqueous solution of the sulphates with  $\text{NaHCO}_3$  in

slight excess, adding a few drops of acetic anhydride, and extracting the solution with  $\text{CHCl}_3$ .

**Acetylsalicylic Acid.** H. L. Smith. (*Pharm. J.*, 1915 [4], 40, 200.) The identity of acetylsalicylic acid with Bayers "Aspirin" is demonstrated. Some commercial specimens contain free salicylic acid, and difference in the rate of solubility of pure specimens is noted. Difference in appearance is probably due to varying methods of crystallization.

**Alcohol, Approximate Determination of, by means of Salting out.** J. Seymour and G. McDermand. (*J. Amer. Pharm. Assoc.*, 1915, 4, 174.) Dry  $\text{K}_2\text{CO}_3$  is added to a known volume of the liquid to be tested, in a closed graduated cylinder, until a saturated solution is obtained. The supernatant layer is then read off as alcohol.

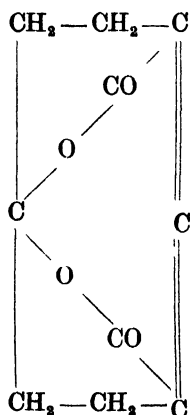
**Alcohol Determination in the Tropics.** K. C. Browning and C. T. Symons. (*J.S.C.I.*, 1914, 33, 819-21.) Comparative EtOH determinations with pycnometer, glass and brass hydrometers and Zeiss' immersion refractometer show that Sikes' brass hydrometer reading at  $30^\circ\text{C}$ . and using the tables supplied by the maker are quite inaccurate, especially at low strengths. Bedford's tables give fair results. Glass hydrometers and Bedford's tables give more satisfactory results.

**Amyl Nitrite, Preparation, Purity and Tests.** F. O. Taylor. (*J. Amer. Pharm. Assoc.*, 1915, 3, 1584.) An exhaustive paper detailing a large number of practical results is thus summarized: The character of amyl nitrite on the American market to-day is, as a whole, mediocre, with a little very good and other small part very bad. With proper care first-class amyl nitrite may be made commercially. The nitrous acid process is preferable for commercial work with fairly pure alcohol, but the sodium nitrite-sulphuric acid process is preferable when very pure alcohol is used and a product of great purity desired. A very pure alcohol seems to be less easily converted to nitrite by the nitrous acid process than one less pure. While the most of an alcohol may be easily changed to nitrite by the nitrous-acid process, the last portions are much more difficult to convert. The amyl nitrites made from the first and last fractions of an alcohol boiling chiefly between  $128^\circ$  and  $132^\circ\text{C}$ . differ slightly in boiling point. Certain compounds of very high boiling point

are formed, and pyridine nitrate is produced by the nitrous-acid process from even a very pure alcohol and to a greater degree from less pure varieties. Amyl nitrite shows a reduction of acidity by distillation. A binary mixture of pure amyl nitrite and water distils at about  $80^{\circ}\text{C}$ ., and this fact may be used as a more delicate test for moisture in amyl nitrite than the freezing method. Neither the assay nor distillation alone give reliable information as to the character of amyl nitrite.

The following is by no means too high a standard : *Sp.g.*—0.870–0.880 at  $15^{\circ}$ —0.865–0.875 at  $25^{\circ}$ . *Acidity*—U.S.P. *Aldehyde*—U.S.P. *Nitrate*—No more than a faint trace when tested by the method given. *Assay*—The U.S.P. method, modified to require not less than 90 per cent. *Boiling Point*—At least 80 per cent. to distil between  $90^{\circ}$  and  $100^{\circ}$ . *Moisture*—No traces of water to be shown at the beginning of distillation by a momentary lowering of the boiling of any part to about  $80^{\circ}\text{C}$ .

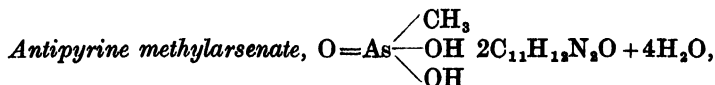
**Anemonin, Constitution of.** Y. Asahina. (*Jap. Pharm. J.*, 1915 [396]; *Chem. Abstr.*, 1915, 9, 1482.) Experimental and physical data which are fully described lead the author to assign the structural formula :



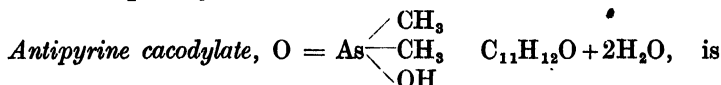
to anemonin.

**Antipyrine Methylarsenate and Cacodylate.** L. Barthe. (*Bull. Soc. pharm. de Bordeaux*; *L'Union pharm.*, 1915, 56, 104, 106.)





is obtained by adding a warm EtOH solution of 1 mol. of methylarsenic acid to 2 mols. of antipyrine in strong EtOH solution. The mixture is boiled for a few minutes, filtered, and set aside under a bell-jar over  $\text{H}_2\text{SO}_4$ . Fine bulky crystals are thus readily obtained; soluble 28.5 : 100 of water at  $20^\circ\text{C}$ . soluble in EtOH; optically inactive.



obtained by a similar process, employing a mixture of alcohol and water as the solvent, and 1 mol. of each of the constituents. It forms crystals which, however, are smaller than those of the methylarsenate. Antipyrine cacodylate is soluble 24.5 : 100 in water at  $12^\circ\text{C}$ . It melts below  $100^\circ\text{C}$ . Both these salts should have valuable therapeutic properties.

**Asphalt, Trinidad.** C. Richardson. (*J. Phys. Chem.*, 1915, **19**, 241; *J.S.C.I.*, 1915, **34**, 413.) The oil sands occurring at different depths in the locality of the mud spring in the island of Trinidad contain a highly asphaltic petroleum, which, upon meeting the fine silica and clay mud of the spring, is emulsified by the action of the natural gas under high pressures, forming the so-called "soft pitch." The "soft pitch" slowly evolves gas and gradually hardens, the process being possibly catalyzed by absorbed ferrous sulphate. The approximate composition of the crude asphalt, which is remarkably homogeneous, is: water and gas, 29.0 per cent.; bitumen soluble in cold  $\text{CS}_2$ , 39.0 per cent.; bitumen absorbed and retained by the disperse mineral matter, 0.3 per cent.; mineral matter on ignition with  $\text{Ca}_3\text{PO}_4$ , 27.2 per cent.; water of hydration of clay, 4.2 per cent. The water obtained from the melted material contains about 20 Gm. of salts per litre, including  $\text{Na}_2\text{SO}_4$ ,  $\text{FeSO}_4$ ,  $\text{Am}_2\text{SO}_4$ ,  $\text{NaCl}$ , and small amounts of iodides and borates. The mineral matter obtained on ignition consists of impalpably fine silica, clay, and the non-volatile salt residue. The preparation from the asphalt, by means of carbon bisulphide and other organic solvents, of a bitumen free from mineral matter is rendered difficult by the presence of colloidal material, consisting mainly of bitumen absorbed by clay and other mineral matter in a

state of high dispersion ; about one-half of the bitumen is soluble in naphtha of B. 88° sp.g. 0.621, and this portion is not absorbed. The crude asphalt thus consists of a suspension of relatively large size mineral particles in an extremely viscous medium, together with highly dispersed mineral matter in colloid form, intimately mixed with an emulsion of a thermal water with the bitumen present. A very similar product results when a crude Bermuda asphalt, practically free from mineral matter, is softened below 100°C. and emulsified with a paste of colloidal clay and water. The presence of suspensoid and dispersoid material in asphalt enhances the surface energy and viscosity and lessens the ductility and susceptibility to change of temperature ; the effect of an added mineral dust, though of the same nature, is far less than that of the highly dispersed colloids which are present in Trinidad asphalt.

**Ascaridolic Acid, Resolution of.** E. K. Nelson. (*J. Amer. Chem. Soc.*, 1914, **36**, 2521.) Theoretically, ascaridolic acid, having the structure of a 1-4-cineolic acid, should be a racemic compound. This has been proved to be the case. By means of cinchonidine, it has been separated into dextro- and laevo-ascaridolic acid. That base forms a sparingly soluble salt with *d*-ascaridolic acid and a salt readily soluble in water with the *l*-acid. Consequently the resolution was easy. The liberated dextro-acid has the  $[\alpha]_D +13.93^\circ$  and the laevo-form the  $[\alpha] -13.77^\circ$ .

**Beta-naphthol, Colour Reaction for.** J. Katayama and B. Ikeda. (*Yakugakuzashi* ; *J. Pharm. Chim.*, 1914, **11**, 73.) One c.c. of very dilute solution suspected to contain  $\beta$ -naphthol is mixed with a few drops of  $H_2SO_4$  and 0.05 c.c. of a 0.01 per cent. solution of  $NaNO_3$  is added. If  $\beta$ -naphthol is present to the extent of 0.01 to 0.001 Gm. in 100 c.c. a violet colour will be formed ; and a distinct violet tint is evident even with a dilution of 0.0002 Gm. of  $\beta$ -naphthol in 100 c.c.

**Calcium Theobromine Compound.** L. Rousseau. (*Comptes rend.*, 1915, **160**, 363.) The compound  $Ca(C_7H_5N_4O_2)_2 \cdot 9H_2O$  is obtained by boiling together in water, protected from  $CO_2$ , 2 mols. of  $C_7H_5N_4O_2$  and 1 mol. of pure  $CaO$ . On cooling, long radiating needles of the above compound separate out. Soluble without dissociation in water at 16°C. 1:16 ;

at 100°C. 1 : 14; very sparingly soluble in alcohol. Decomposed by  $\text{CO}_2$  and weak acids, liberating colloidal  $\text{C}_7\text{H}_5\text{N}_4\text{O}_2$ . To this fact the prompt and active diuretic effect of the compound is due, the colloidal base being liberated by the acids of the gastric secretion.

**Chloroform, Test of the Italian Pharmacopœia for the Presence of Aldehyde in.** R. Pajetta. (*Boll. Chim. Farm.*, 1914, 53, 161; *Chem. Abstr.*, 1915, 9, 1367.) The Italian pharmacopœia provides that  $\text{CHCl}_3$ , in order to meet the requirements, shall not assume a yellow colour when 5 c.c. is placed in contact with a piece of KOH and that the latter shall remain white for the period of 12 hours (absence of aldehydes). Each of several samples of the best commercial  $\text{CHCl}_3$  obtainable was washed several times with  $\text{H}_2\text{O}$ , dried and distilled and divided into three portions: (a)  $\text{CHCl}_3$  alone, (b)  $\text{CHCl}_3$  with 0.5 per cent. absolute EtOH (Italian pharmacopœia) and (c)  $\text{CHCl}_3$  with 1.0 per cent. absolute EtOH (as above). The above test for the presence of aldehydes was applied in identical manner to each of the three portions. (a) remained unchanged indefinitely, while the KOH in (b) and (c) gradually assumed the characteristic yellowish red tint; the presence of aldehyde and AcOH (as acetate) was afterward independently confirmed in (b) and (c), although these substances were absent in (a). This indicates that aldehyde is produced by the action of KOH on the added EtOH. The requirements of the Italian pharmacopœia are therefore inconsistent in that the addition of 0.5–1.0 per cent. EtOH to  $\text{CHCl}_3$  is allowed, while a test which permits the oxidation of EtOH to aldehyde is prescribed for the detection of the latter in  $\text{CHCl}_3$ . When EtOH is present in  $\text{CHCl}_3$ , the test for aldehydes should be made in a different manner, e.g. with a warm solution of KOH or of pure  $\text{AgNO}_3$  as prescribed by the French pharmacopœia.

**Cholesterol Contents of Human and Animal Brains.** Mary C. Rosenheim. (*Biochem. J.*, 8, 82; *Chem. Abstr.*, 1914, 8, 3071.) Rosenheim's method for the preparation of cholesterol and Windaus's method for its quantitative estimation were used. The percentages of cholesterol in dry brain are: Man (a) 9.22, (b) 9.01, child (aged 3 months) 4.89, child (aged 5 days) 5.29, foetus (aged 36 weeks) 4.07, dog 11.59, cat 9.99, ox (a) 11.28, (b) 12.04, sheep 10.37, rabbit (a) 9.57, (b) 9.11, fowl 7.4, codfish (a) 12.02, (b) 11.89.

**Chrysarobin, Commercial.** R. Eder. (*Arch. Pharm.*, 1915, 253, 1-33; *J.S.C.I.*, 1915, 34, 681.) Commercial chrysarobin after oxidation by air in alkaline solution was found to consist of about 26 per cent. of amorphous dark violet and brownish red products, 18 per cent. of dehydro-emodin-anthranol monomethyl ether, 2 per cent. of emodin, and 32 per cent. of a mixture composed of 71 per cent. of chrysophanic acid and 29 per cent. of emodin monomethyl ether. Dehydro-emodin-anthranol monomethyl ether is also present in the original chrysarobin, which contains, in addition, chrysophanic acid, emodin monomethyl ether, and emodin, either wholly or partly in a reduced form. The presence of dichrysarobin methyl ether, mentioned by Jowett and Potter (*Y.B.*, 1904, 52), is considered very improbable.

**Citric Acid, New Colour Reaction for.** E. P. Häussler. (*Chem. Zeit.*, 1914, 38, 937; *Chem. Abstr.*, 1915, 9, 488.) When a water-EtOH solution of vanillin containing citric acid is evaporated to dryness, and a small amount of 25 per cent.  $\text{H}_2\text{SO}_4$  added, and warmed 10-15 minutes, a strong violet colour is produced. This dissolves in  $\text{H}_2\text{O}$  to give a green solution, which turns red when AmOH is added, and even in very dilute solutions is characteristic of citric acid. This reaction is not produced by acetic, malic, oxalic, malonic, benzoic, salicylic, lactic, or tartaric acids. Five c.c. of a 1 per cent. citric acid solution, tested by this reaction, gave a strong colour to 1.5 litres  $\text{H}_2\text{O}$ , but with 1 c.c. there were no definite results. Sugars and albuminous substances interfere with the reaction, but these may be removed by  $\text{Pb C}_2\text{H}_3\text{O}_2$  and EtOH.

**Collargol and Protargol, Valuation of.** (*Supplement to Ph. Ned. IV.*; *J. Pharm. Chim.*, 1915, 11, 239.) Collargol should contain from 75 to 80 per cent. of Ag. Collargol, 0.125 Gm., is dissolved in exactly 25 c.c. of distilled water; after standing 3 hours, 20 c.c. of the clear liquid is pipetted off, and treated with 30 c.c. of  $\text{HNO}_3$ . The mixture is heated for 5 minutes and cooled. Two c.c. of  $\text{H}_2\text{SO}_4$  is then added and the heating is repeated until no more acid fumes are evolved. The residual liquid is cooled, 100 c.c. of distilled water added, and 1 c.c. of saturated solution of ferric sulphate. The Ag is then titrated with N/10 AmCNS solution. From 6.9 to 7.4 c.c. should be required to give a permanent pink tint: 1 c.c. of the solution

=10.8 Mgm. of Ag. In the case of *protargol*, 1 Gm. is ashed; the residue is dissolved in  $\text{HNO}_3$ ; the solution made up to 100 c.c., then titrated with  $\text{N}/10$  AmCNS as described above.

**Coumarin, Detection of, in Small Amounts, in Factitious Vanilla Extracts.** J. R. Dean. (*J. Ind. Eng. Chem.*, 1915, 7, 510.) The following modification of Wichmann's method is specially suitable for detecting small amounts of coumarin in presence of vanillin, such as occur in commercial vanillin extracts. The original method depends on the conversion of coumarin into salicylic acid; hence that acid, or saccharin, must be removed before applying the test. This is attained as follows: Render a de-alcoholized portion of the extract alkaline with 5 c.c. of AmOH (use of the residue after an alcohol determination is recommended) and extract with 15 c.c. of  $\text{Et}_2\text{O}$ . Vanillin, salicylic acid and saccharin are insoluble in  $\text{Et}_2\text{O}$  in the presence of AmOH, while coumarin is readily dissolved. Transfer the  $\text{Et}_2\text{O}$  to a nickel or porcelain crucible and evaporate off the solvent. Add 5 drops of a 50 per cent. solution of KOH and, after carefully drying, fuse at the lowest possible temperature, care being taken to avoid any blackening. Dissolve the mass in a few c.c. of water, render acid with dilute  $\text{H}_2\text{SO}_4$  and transfer to a test tube. Add 5 c.c. of  $\text{CHCl}_3$  to dissolve out the salicylic acid produced in the fusion and shake the tube vigorously. Allow the  $\text{CHCl}_3$  to separate and remove it with a small pipette extended to the bottom of the tube. Transfer it to a second test tube, filtering through a small plug of cotton. Add 1 or 2 c.c. of water, containing a drop or two of  $\text{Fe}_2\text{Cl}_6$  solution, and shake as before. The presence of coumarin is indicated by the formation of the purple colour of ferric salicylate.

**Crystals of Organic Compounds Coloured Blue by Iodine.** G. Barger and W. W. Starling. (*Proc. Chem. Soc.*, 1914, 30, 2-3.) The blue organic compounds formed from I may be either amorphous or crystalline; the former are typical examples of absorption. The blue crystals may be regarded as solid solutions of I in the organic compounds, not necessarily in stoichiometrical proportions. They result only when the crystal is formed in the presence of I, either from a saturated solution containing I or by sublimation in its presence. Colourless crystals of the substances, when once formed, cannot take up I. Narceine, for example, when dissolved in pyridine,

together with I, gives rise to blue crystals when light petroleum is added. All of the crystals examined so far are more or less strongly pleochroic; no connexion has been found between the crystalline form and the tendency to form these "mixed" crystals with I. The additive compounds may be analogous to oxonium compounds, but since they are also given by substances not containing a pyrone nucleus, they might possibly show a greater analogy to the K additive compounds of ketones described by Schlenk and Thal.

**Diphenylsemicarbazide as a Reagent for the Detection of Carbonyl Derivatives.** B. Toschi and A. Angiolani. (*Gaz. Chim. ital.*, 1915, 45, I, 205; *J.S.C.I.*, 1915, 34, 572.) 4·4'-Diphenylsemicarbazide, obtained in almost quantitative yield by mixing EtOH solutions of equivalent quantities of diphenylurea chloride and hydrazine hydrate, forms an excellent reagent for identifying compounds containing the carbonyl group. It possesses the advantages over semicarbazide that it does not decompose on exposure to air and light and that the diphenylsemicarbazones are less soluble than the corresponding semicarbazones. It reacts readily even with acetone, salicylaldehyde, benzophenone, and certain sugars which react only slowly with semicarbazide. In the case of aliphatic carbonyl derivatives it is preferable to use the hydrochloride of diphenylsemicarbazide; with aromatic compounds the free base is used. The diphenylsemicarbazones of the following compounds are described: acetone, colourless needles, m.p. 119°C.; acetaldehyde, silky needles, m.p. 153°C.; cænanthol, colourless needles, m.p. 133°-134°C.; ethyl acetoacetate, m.p. 103°-104°C.; dextrose, white needles containing 1 mol. H<sub>2</sub>O, m.p. 164°-166°C.; cinnamic aldehyde, yellow needles, m.p. 164°-166°C.; cumic aldehyde, m.p. 162°C.; salicylaldehyde, colourless needles, m.p. 209°C.; vanillin, white needles, m.p. 180°-181°C.; piperonal, yellow needles, m.p. 173°C.; benzophenone, white needles, m.p. 186°-187°C.; citronellal, white crystals, m.p. 109°-110°C.; camphor, silky needles, m.p. 154°-155°C.

**Ether, Commercial, Examination of.** B. S. Ellis and A. Flett. (*Pharm. J.*, 1915 [4], 40, 387.) It is not possible to obtain concordant results in sp.g. determination of Et<sub>2</sub>O when the ordinary sp.g. bottle with the perforated stopper is used. The hydrometer, the "bead" method and the closed

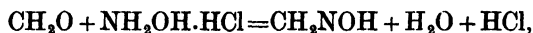
sp.g. give better results. The "head" method is to be preferred. The mere determination of sp.g., without quantitative fractional distillation, is valueless. Commercial ethers obtained by the authors required fractional distillation to bring them to the B.P. 1914 standard.

**Ethyl Alcohol, Specific Reaction of.** A. Toninelli. (*Ann. Chim. anal. Appl.*, 1914, 19, 169; *Analyst*, 1914, 39, 319.) Two c.c. of the distillate under examination is shaken in a stoppered tube with 2 c.c. of a solution of 12 Gm. in  $\text{Et}_2\text{O}$  100 c.c., and then, after 2 minutes, with 4 c.c. of KOH solution (4:10) until decolorized. It is then tested with 2 c.c. of a solution of 1.5 Gm. of pure dinitrotoluene in 200 c.c. of a mixture of 1 part of  $\text{CS}_2$  and 2 parts of  $\text{Et}_2\text{O}$ . In the presence of  $\text{EtOH}$  (3 per cent. and over) the upper layer, which separates after the shaking, assumes an orange colour, which gradually fades, and then becomes red. Methyl alcohol, acetone, and aldehyde do not interfere with the reaction, but similar colorations are given by certain higher alcohols. If the presence of such be suspected, from 25 to 100 c.c. of the sample is repeatedly shaken with twice the volume of a 5 per cent. solution of alum, and a little  $\text{C}_6\text{H}_6$  or petroleum spirit. The aqueous layer is drawn off and fractionated, and the portions distilling between  $60^\circ$  and  $80^\circ\text{C}$ . tested as described. If insufficient distillate is obtained, 3 c.c. of pure acetone is added, and the distillation repeated.

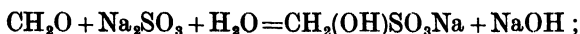
**Formaldehyde, Hexamethylenetetramine and Formalin, Determination of, in Tablets.** W. Stuewe. (*Arch. Pharm.*, 1914, 252, 430.) When formaldehyde is added to a solution of  $\text{HgCl}_2 + \text{KI}$  and then KOH is added, an immediate reduction to metallic Hg takes place,  $\text{CH}_2\text{O} + \text{K}_2\text{HgI}_4 + 3\text{KOH} = \text{Hg} + \text{HCO}_2\text{K} + 4\text{KI} + 2\text{H}_2\text{O}$ . The reaction is applied to the determination of  $\text{CH}_2\text{O}$  as follows: Dissolve 1 Gm. of  $\text{HgCl}_2$  in 20 c.c. of water, add 0.5 Gm. of gum arabic and 3 Gm. of KI, then agitate until solution results. Now add 10 c.c. of 15 per cent. NaOH and 10 c.c. of a solution of 1 c.c. of  $\text{CH}_2\text{O}$  in 100 c.c. of water, then acidify after 1 minute with 20 c.c. of dilute  $\text{H}_2\text{C}_2\text{H}_3\text{O}_2$ , place the container several minutes in cold water, dissolve the precipitated Hg in 25 c.c. N/10 I and titrate the I excess with N/10  $\text{Na}_2\text{S}_2\text{O}_3$ . One c.c. I solution = 0.0015 Gm.  $\text{CH}_2\text{O}$ . Hexamethylenetetramine: Heat 0.5 Gm.  $\text{C}_6\text{H}_{12}\text{N}_4$  with a mixture of 100 c.c. water and 10 c.c. 25 per cent. HCl

15 minutes on a water-bath in a flask provided with an air-cooled tube, allow to cool, wash out the tube with water, finally diluting the liquid to 250 c.c. Treat 10 c.c. of this solution (=0.02 Gm. of sample) as above. One c.c. N/10 I=0.001167 Gm. hexamethylenetetramine. Formalin tablets: Into the Nessler reagent prepared as above introduce 0.025–0.03 Gm. of the powdered sample and proceed as for  $\text{CH}_2\text{O}$ . By using N/100 I solution very minute quantities of  $\text{CH}_2\text{O}$  may be detected.

**Formaldehyde Solution, Determination of Formaldehyde and Methyl Alcohol in.** G. Lockemann and F. Croner. (*Zeits. Analyt. Chem.*, 1915, **54**, 11; *Analyst*, 1915, **40**, 237.) In an aqueous solution containing both formaldehyde and  $\text{HCHO}_2$ , the former may be estimated by adding hydroxylamine hydrochloride and titrating the liberated  $\text{HCl}$ , or by adding  $\text{Na}_2\text{SO}_3$  and titrating the free alkali formed; the methyl alcohol is estimated by oxidizing the solution with  $\text{KMnO}_4$  and calculating the quantity of alcohol present from the amount of  $\text{KMnO}_4$  reduced, allowance being made for the  $\text{KMnO}_4$  reduced by the formaldehyde. In the hydroxylamine method the reaction proceeds according to the equation



and methyl-orange is used as the indicator in titrating the acid produced. The reaction with  $\text{Na}_2\text{SO}_3$  is shown by the equation



rosolic acid is used as the indicator in the titration, since it gives a sharper end-reaction than does phenolphthalein. For the oxidation with permanganate, the formalin (40 per cent. formaldehyde solution) is diluted with 100 times its volume of water, and 5 c.c. of this solution is mixed in a flask with 75 c.c. of water and 25 c.c. of alkaline N/2  $\text{KMnO}_4$  solution (15.82 Gm. of  $\text{KMnO}_4$  and 40 Gm. of  $\text{NaOH}$  per litre); the mixture heated for 20 minutes on a water-bath, then treated with an excess of N/2 oxalic acid solution (31.51 Gm. of crystallized oxalic acid and 75 c.c. of concentrated  $\text{H}_2\text{SO}_4$  per litre), and the excess of oxalic acid is titrated with acid N/2  $\text{KMnO}_4$  solution (containing 40 Gm. of crystallized  $\text{H}_3\text{PO}_4$  per litre). One c.c. of N/2  $\text{KMnO}_4$  solution is equivalent to 0.00375 Gm. of formaldehyde or 0.00267 Gm. of methyl alcohol. The authors found that it was impossible to separate the aldehyde from the



alcohol by distillation even after the addition of substances such as AmOH, alkali bisulphite, the sodium salt of sulphanilic acid, etc.; traces of formaldehyde were always found in the distillate. Attempts were also made to bring about the separation by converting the formaldehyde into an insoluble compound by treatment with aniline or *p*-nitrophenylhydrazine and subsequent filtration, but it was not possible, by distillation, to separate the methyl alcohol from the excess of precipitant in the filtrate.

**Galls of *Quercus Aegliops*, Nitrogenous Constituent of.** M. Nierenstein. (*Zeits. Physiol. Chem.*, 1914, **92**, 53.) The  $C_6H_6$  and  $CCl_4$  extracts from the galls of *Quercus aegliops*, after distilling off the solvents, and standing for  $2\frac{1}{2}$  years, were found to have deposited crystals. These were recrystallized from EtOH as prismatic needles; m.p.  $234^\circ$ – $238^\circ C.$  with decomposition;  $[\alpha]_{D_{16}} -57.35^\circ$ . This was identified as 1-gallol-leucine  $Me_2CHCH_2CH(COOH)NHCOC_6H_4(OH)_3$  giving gallic acid and racemic leucine on hydrolysis.

**Glycerin, Detection and Determination of, Free and Combined.** M. François and E. Boismenu. (*J. Pharm. Chim.*, 1915, **11**, 49.) Free glycerin, after having been freed by heating on the water-bath from formaldehyde, alcohol, water and other volatile impurities may be detected by heating it to decomposition with  $KHSO_4$ , and the red colour formed by the acrolein vapours so formed on contact with rosaniline bisulphite solution. This red colour is changed to blue on warming. Glycerin combined in glycerophosphates may be detected by the same test, and probably that in lecithin also. The test is thus applied to liquids. The residue obtained by evaporation on the water-bath is extracted with EtOH-Et<sub>2</sub>O mixture. This extract is again evaporated on the water-bath. If much glycerin is present, one or two drops of the liquid residue is allowed to fall on 5 Gm. of  $KHSO_4$ , placed in a test-tube fitted with a tube condenser and then heated in a flame. The vapour thus obtained is passed over the surface of 5 c.c. of decolorized rosaniline bisulphite reagent. In presence of glycerin a red colour will be formed in 30 seconds. On plunging this in a boiling water-bath, the red tint will change to blue in 30 seconds. If only a small amount of glycerin is present, the residue of extraction will be pasty. This is rubbed down with the 5 Gm. of  $KHSO_4$  and treated as above. Solid substances, such as

glycerophosphates, may be mixed with the  $\text{KHSO}_4$  and tested directly. The determination of glycerin may be performed by Hehner's method, or Martin's modification thereof, provided that a sufficient excess of  $\text{K}_2\text{Cr}_2\text{O}_7$  and  $\text{H}_2\text{SO}_4$  is employed, and oxidation is performed at the boiling point of the mixture. In Martin's original method, oxidation is not complete. The details of the improved method are as follows: Twenty-five c.c. of a solution containing approximately 0.05 Gm. of glycerin is introduced into a 250 c.c. boiling flask with 25 c.c. of standard  $\text{K}_2\text{Cr}_2\text{O}_7$  solution, containing 74.558 Gm. per litre, and 20 c.c. of a mixture of equal parts by weight of  $\text{H}_2\text{SO}_4$  and water. After boiling for 2 hours under a reflux condenser, 50 c.c. of water is distilled off, and the amount of unreduced  $\text{K}_2\text{Cr}_2\text{O}_7$  remaining in the cold residue is titrated by means of standard ferrous ammonium sulphate solution. Each c.c. of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution used up = 0.01 Gm. of glycerin. In the case of a solid glycerophosphate, the above method is followed, using approximately 0.250 Gm. of the sample which is mixed with 25 c.c. of the  $\text{K}_2\text{Cr}_2\text{O}_7$  solution and treated as described above.

**Glycerin, Determination of, in Galenicals.** C. H. Briggs. (*J. Amer. Pharm. Assoc.*, 1915, 4, 75.) The method consists in distilling the glycerin, *in vacuo*, in the presence of excess of sandalwood oil, which carries over the whole of the glycerin present and separating the latter in the distillate. Sufficient of the sample to obtain about 2 Gm. of glycerin is taken and placed in a 500 c.c. side neck distillation flask with 0.5 Gm.  $\text{MgO}$ . Warm on a steam-bath for 5 minutes. Now add 75 c.c. of santal oil and distil *in vacuo* until about two-thirds of the oil has been distilled. Rinse the condenser with about 100 c.c. of purified petroleum benzin and add to the distillate. Now rinse the condenser well with 5 c.c. of water and add to the distillate. Transfer the mixed distillate and washings to a separator, shake out and draw off the aqueous layer to a second separator. Extract the benzin oil solution three times with 5 c.c. of water to completely remove the glycerin and add to the first aqueous extract. Shake the combined aqueous extracts with 30 c.c. of petroleum benzin to remove traces of oil. Allow to stand half an hour and draw off the aqueous layer into a tared four-inch Petrie dish. Rinse the separator with 5 c.c. of water and add to the glycerin extract. Evaporate off most of the water at a low temperature (not over  $50^\circ\text{C}$ .)

and dehydrate in a vacuum dessicator over  $\text{H}_2\text{SO}_4$  for 24 hours or to constant weight. This anhydrous glycerin is very hygroscopic and must be weighed quickly. To convert to ordinary commercial glycerin divide the weight obtained by 0.97. The distillation must be carried out cautiously at first to prevent bumping. After the water has passed over, the distillation proceeds quietly and can be carried out rapidly. A free flame should be used and the burner should be held in the hand and kept in constant rotation round the bottom of the flask.

**Glycerin, Determination of, in Tablets and Confections.** L. Forman. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1644.) Enough tablets are taken to weigh about 5 Gm., these are dissolved in water, evaporated to syrupy consistence, 15 c.c. of milk of lime added and the mixture evaporated to a thick paste, stirring frequently to prevent it from drying hard on sides of dish. This mass is then rubbed to a smooth paste with 5 c.c. of water, 45 c.c. absolute EtOH is added, heated to incipient boiling, and heavy particles allowed to settle. The supernatant liquid is then transferred to filter, and dish and filter washed with 95 per cent. EtOH until the filtrate measures 150 c.c. This is evaporated on a water-bath at  $85^\circ\text{C}$ . to a syrupy consistence. This residue is taken up with 10 c.c. absolute EtOH, transferred to 50 c.c. graduated cylinder and the dish washed with 2 c.c. portions of absolute EtOH and transferred to the cylinder; then 30 c.c. of anhydrous  $\text{Et}_2\text{O}$  are added in 10 c.c. portions and shaken thoroughly after each addition. This is allowed to stand until perfectly clear, then decanted through a dry filter, and the cylinder and filter washed with 25 c.c. of EtOH- $\text{Et}_2\text{O}$  mixture in above proportions. This is then evaporated to 5 c.c., 20 c.c. of water added and evaporated to 5 c.c., 10 c.c. water added and again evaporated to 5 c.c. This is transferred to a 50 c.c. volumetric flask, the beaker washed with hot water, then freshly precipitated  $\text{Ag}_2\text{CO}_3$  (0.1 Gm. of  $\text{Ag}_2\text{SO}_4$ , plus excess of  $\text{Na}_2\text{CO}_3$ ) is added. The mixture shaken frequently during 10 minutes, then 0.5 c.c. basic lead acetate is added and again shaken frequently during 10 minutes and made up to mark. Twenty-five c.c. of the filtrate is placed in a 250 c.c. flask, 1 c.c. concentrated  $\text{H}_2\text{SO}_4$  added, to precipitate excess of Pb, then 30–40 c.c. strong  $\text{K}_2\text{Cr}_2\text{O}_7$  solution (7.5 Gm. of  $\text{K}_2\text{Cr}_2\text{O}_7$  and 15 c.c. concentrated sulphuric acid per 100 c.c.), and 24 c.c.  $\text{H}_2\text{SO}_4$ , and the mixture placed in boiling

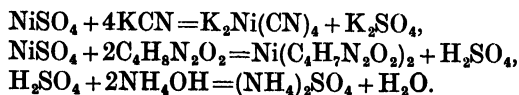
water-bath for 25 minutes. Make up to mark, cool, take aliquot of 20 c.c., dilute with 50–70 c.c. water and titrate excess of bichromate with ferrous ammonium sulphate solution (30 Gm. per litre and 50 c.c.  $\text{H}_2\text{SO}_4$ ). Standardize ferrous ammonium sulphate solution against a 1–20 dilution of  $\text{K}_2\text{Cr}_2\text{O}_7$  solution, then calculate glycerin, by finding excess of  $\text{K}_2\text{Cr}_2\text{O}_7$  in oxidized glycerin solution. The number of cubic centimetres of strong  $\text{K}_2\text{Cr}_2\text{O}_7$  added, minus excess found after oxidization, multiplied by 0.01 Gm. equals weight of glycerin in the 25 c.c. purified solution used. This multiplied by two gives total weight of glycerin.

**Glycerophosphates, Determination of Phosphorus in.** M. François and E. Boismenu. (*J. Pharm. Chim.*, 1914, [7], 11, 65.) Approximately 0.500 Gm. of the sample, previously dried at  $150^\circ\text{C}$ ., accurately weighed, is mixed in a 150 c.c. flask with 10 Gm. of  $\text{H}_2\text{SO}_4$ , 10 Gm. of water, and 4 Gm. of  $\text{K}_2\text{Cr}_2\text{O}_7$ , added in 3 portions. A brisk reaction ensues, with evolution of  $\text{CO}_2$ . The flask is then attached to an upright condenser, and the contents are boiled for 2 hours. Without cooling, the liquid is transferred to a 500 c.c. flask, the smaller flask being washed out with 30 c.c. of water, and the whole made up to 500 c.c. To 50 c.c. of the liquid thus obtained, 10 Gm. of crystalline  $\text{Na}_2\text{SO}_3$  is added in 3 portions, to reduce all the  $\text{K}_2\text{Cr}_2\text{O}_7$  to the state of the green sulphate. Twenty Gm. of  $\text{NaNO}_3$  is then added, and solution brought about on the water-bath. Finally 300 c.c. of molybdic reagent is added and the mixture is kept on the water-bath for 2 hours. It is then left for 24 hours at the normal temperature, when the precipitate is collected and treated in the usual manner.

**Hydrocyanic Acid in Small Amounts, Colorimetric Determination of.** A. Viehovever and C. O. Johns. (*Amer. J. Pharm.*, 1915, 87, 261.) The distillate, or similar aqueous liquid containing a small amount of free HCN, is treated with 0.02 to 0.1 Gm. of NaOH and concentrated in a round bottom flask of 200 c.c. capacity on a water-bath kept below  $70^\circ\text{C}$ ., using a vacuum pump and condenser. To avoid any loss by spattering, the flask is fitted to the condenser by means of a splash trap. Concentration is carried to less than 1 c.c. of liquid residue. Two-tenths to 0.5 c.c. of 3 per cent. freshly-prepared  $\text{FeSO}_4$  solution and about 0.05 Gm. of KF are then

added. The flask is exhausted at once by means of a water vacuum pump. The contents are mixed by rotating. After 5 to 10 minutes the flask is detached from the pump and the mixture acidified with 30 per cent.  $\text{HNO}_3$ . The blue colour appears at once. Where only traces of  $\text{HCN}$  are present it is sometimes necessary to warm to about  $50^\circ\text{C}$ . in a water-bath before the colour appears. The suspension is then diluted to a volume that would give a colour density convenient to compare with a suspension of Prussian blue made from a known weight of  $\text{KCN}$ . As a standard a suspension of Prussian blue made from 1 Mg. of  $\text{KCN}$  may be used. Such a suspension diluted to 25 c.c. gives a colour of convenient density. For comparison a Duboscq colorimeter may be employed. If the cyanide solution to be tested is sufficiently concentrated so that further evaporation is unnecessary, the test may be made in a test-tube. Air may be kept out by means of a stopper and the tube rotated only enough to mix the reagents, allowing the mixture to stand for 5 to 10 minutes before acidifying. Much shaking must be avoided to prevent excessive oxidation of the  $\text{Fe}(\text{OH})_2$ . The test is applicable microchemically to sections of cyanogenetic plant tissues.

**Hydrocyanic Acid, New Method for Determining.** G. E. F. Lundell and J. A. Bridgman. (*J. Ind. Eng. Chem.*, 1914, 6, 554.) The method consists in titrating an ammoniacal cyanide solution containing a small quantity of dimethyl glyoxime, with a standard nickel ammonium sulphate solution until a permanent red precipitate is produced. The reactions involved are expressed by the equations :



No permanent red precipitate of nickel dimethyl glyoxime is formed until all of the cyanide has been used up in the reaction expressed by the first equation. The ammoniacal cyanide solution is used, since free  $\text{H}_2\text{SO}_4$  hinders the precipitation of nickel dimethyl glyoxime.

*Solutions Required.*—1. *Standard nickel solution*, prepared by dissolving 15.3 Gm. of nickel ammonium sulphate in water containing 2 c.c. of concentrated  $\text{H}_2\text{SO}_4$ , diluting to 1 litre,

and standardizing as directed below. 2. *Dimethyl glyoxime solution*, prepared by dissolving 8.9 Gm. of dimethyl glyoxime in 1 litre of 95 per cent. alcohol.

*Standardization of Nickel Solution.*—Unless the percentage purity of the nickel ammonium sulphate is known, the prepared nickel solution must be standardized as follows: 25 c.c. portions are diluted with distilled water to 200 c.c., treated with 0.2 Gm. of tartaric acid, and heated to boiling. Glyoxime solution sufficient to precipitate all of the Ni is then added. If the glyoxime solution has been made up according to the formula above, 30 c.c. should be sufficient. After the addition of the glyoxime, the solution is made slightly alkaline with AmOH, boiled for 2 minutes, and then set aside to digest for half an hour. The precipitate is caught on a tared Gooch crucible, washed with 200 c.c. of hot water, dried for 45 minutes at 120°C., and weighed. The weighed precipitate contains 20.31 per cent. Ni. From the equations given above the HCN acid or the KCN titre of the solution can readily be calculated. If a chemically pure KCN is at hand, the above titres can be determined directly by titrating weighed portions as directed in the "Method of Analysis" which follows.

*Method of Analysis for Alkali Cyanides.*—Five Gm. of the sample is dissolved in water and diluted to exactly 500 c.c. Pipetted 50 c.c. portions of this solution are diluted with an equal volume of water, treated with 1 c.c. of AmOH and 0.5 c.c. of the dimethyl glyoxime solution, and then titrated with the standard nickel solution until a permanent red precipitate is produced. The colour play towards the end of the reaction resembles the methyl orange end-point observed in titrating an alkaline solution with an acid solution. The process is considered to be more accurate than the Liebig method, and is specially suitable to the titration of *Aqua Laurocerasi* and *Aqua amygdalae amarae*. If more than 0.5 c.c. of glyoxime is used, the end-point shows a tendency to appear too soon unless the addition of the standard solution is slow and the agitation of the solution very brisk. The cyanide dilution may be varied without serious effect; however, the method works better when the volume is approximately 100 c.c. A large excess of AmOH delays the end-point; 1 to 5 c.c. in the volume specified does no harm. In titrating solutions which contain HCN, a measured volume of solution is made alkaline with ammonium hydroxide and then treated as above.

**Melting Points, Method for Determining.** R. Romanelli. (*Giorn. farm. chim.*; *Répertoire de Pharm.*, 1915, 27, 154.) A thin Pt wire about 0.3 or 0.4 mm. diameter is formed into a loop of 8 or 9 mm. diameter. The other end is twisted into a spiral to clasp the bulb of a thermometer. The loop is plunged into a little of the melted material, which is then allowed to congeal, so as to form a thin disc or pellicle between the loop. The thermometer carrying the wire attached is then introduced into the cold bath, and the melting point determined, in the usual manner, when the pellicle breaks. .

**Mercury Haemoglobin Compounds.** R. Robert. (*Apoth. Zeit.*, 1914, 29, 887.) From experiments with various Hg salts and haemoglobin derived from the horse, it is found that the amount of Hg combined varies very markedly with the salt employed. When  $\text{HgSO}_4$  is used the mercury-haemoglobin compound contains 1.7 per cent. of Hg. With  $\text{HgNO}_3$  the percentage is 7; with  $\text{HgC}_2\text{H}_3\text{O}_2$ , 8 per cent.; with  $\text{HgCl}_2$ , 9.5 per cent. The Hg is in very complex combination and almost dissimulated. The corrosive action of ordinary Hg salts was greatly modified in the haemoglobin compounds; but when administered to dogs, Hg could be detected in the urine, and in the viscera and lymphatics, thus showing that partial resorption had occurred. In the case of Cu and Zn salts and haemoglobin, a similar variation in the amount of metal present in the ultimate haemoglobin compound occurred when different salts were used in its preparation.

**Methyl Alcohol, Detection of, in Spirituous Preparations.** — Rinck. (*Zeits. Untersuch. Nahr. Genussmit.*, 28; *Schweiz. Apoth. Zeit.*, 1914, 52, 732.) The liquid to be tested is introduced into a 100 c.c. Erlenmeyer flask to which is fitted a quartz glass tube about 12 mm. wide and 500 mm. long, bent to an angle of  $60^\circ$  about 20 to 30 mm. from the middle. A copper spiral is inserted in the longer bend and heated to redness by means of a Teclius burner. The alcohol is then slowly distilled over the metal and the first portion of the distillate collected in a small ice-cooled flask. One c.c. of this distillate is then tested, in the usual manner, with 5 c.c. of strong  $\text{H}_2\text{SO}_4$  and morphine. A minute trace of  $\text{CH}_3\text{OH}$  may be detected by this method, with certainty.

**Nitrogen Determination by a Modification of Kjeldahl's Method.**

M. Wunder and O. Lascar. (*Annales Chim. analyt.*, 1914, 19, 330.) Three Gm. of oxalic acid, 2 Gm. of  $\text{Na}_2\text{C}_2\text{O}_4$  and 0.5 Gm. of pure  $\text{V}_2\text{O}_5$  are introduced into a 300 c.c. Jena flask. The  $\text{V}_2\text{O}_5$  must be thoroughly calcined, since the commercial oxide may contain notable quantities of  $\text{NH}_3$  which would vitiate the results. If solid, the substance to be examined is previously dried to constant weight. For material containing 5 per cent. of N a minimum weight of 0.6 Gm. is taken with corresponding less weights for material richer in N. This is weighed and introduced into the reaction flask in a small, thin glass capsule about 10 mm. in diameter and 15 mm. deep, which should not weigh more than 1.5 Gm. Volatile liquids may be enclosed in thin glass ampoules, which are broken after introduction into the flask. The weighed material is then introduced into the reaction mixture in the flask, and a mixture of 5 c.c. of  $\text{H}_3\text{PO}_4$  sp.g. 1.71, and 25 c.c. of strong  $\text{H}_2\text{SO}_4$  is added, the flask being kept cool under a stream of water during this addition. The mixture is then gradually heated, with occasional agitation, until organic matter has been completely destroyed. The liquid frequently becomes green at once; but this does not indicate the termination of the reaction. The colour should be allowed to change from black to brown, and finally from reddish-brown to yellowish-green. The solution is then cooled and 100 to 150 c.c. of water is added, the flask being kept cool under the tap. From 0.5 to 0.75 of fine pure iron wire is added, and the liquid is gently warmed for about 30 minutes. It is then transferred to a 1250 c.c. distilling flask, the smaller flask being carefully washed out and the washings added to the larger bulk. The distilling flask is then fitted to a tap funnel containing strong  $\text{NaOH}$  solution and attached to an efficient Kjeldahl condenser. The  $\text{NaOH}$  solution is then run in in strong excess and distillation conducted into a receiver containing a known volume of  $\text{N}/5$   $\text{H}_2\text{SO}_4$ . The process is allowed to proceed slowly for 2 hours. If bumping occur, another 100 c.c. of water is run in through the funnel, and distillation continued. The distillate is then titrated in the usual manner with  $\text{N}/5$   $\text{NaOH}$ . From the amount of acid combined with the  $\text{NH}_3$  evolved, the quantity of N is calculated. In the case of a substance in which the N is combined with O 2 or 3 Gm. of  $\text{Na}_2\text{C}_2\text{O}_4$  or of  $\text{NaCHO}_2$  and 0.75 Gm. of pure Fe wire must be added to the reaction mixture at the commencement of the destruction of organic matter. After this another



0.5 Gm. of Fe must be used. It is important that the first part of the process should proceed slowly.

**Nitroglycerin, Determination of, in Tablets or Triturates.** (*J. Amer. Pharm. Assoc.*, 1915, 4, 219.) Two alternative methods are recommended, a modification of that of Scoville, or of Hay.

*Preparation of Sample (Thorburn Modification).*—Disintegrate a known quantity of tablets or trituration containing about 0.0162 Gm. ( $\frac{1}{4}$  grain) of nitroglycerin in 20 to 25 c.c. of water. Extract four times with successive portions of 25 c.c. of  $\text{Et}_2\text{O}$ . Combine  $\text{Et}_2\text{O}$  extracts and make up to 100 c.c. in a volumetric flask. Then proceed by either of the following methods.

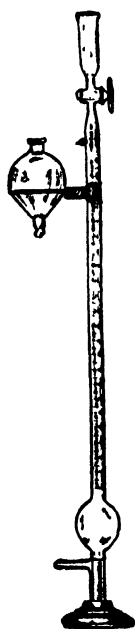
*Estimation by the Modified Scoville Method.*—Of the above  $\text{Et}_2\text{O}$  solution place 40 c.c. in a carefully dried and tared 50 c.c. beaker. (A second aliquot of 20 c.c. may be used as a check.) Evaporate the solvent in a vacuum desiccator. Apply the vacuum gradually so as to prevent ebullition. Leave the beaker in the vacuum 30 minutes after the  $\text{Et}_2\text{O}$  has evaporated. Weigh and calculate  $\text{Et}_2\text{O}$  extract per tablet. Treat the residue with 2 c.c. phenoldisulphonic reagent, rotating the beaker in such a way that the reagent comes into contact with the entire inner surface. After 10 minutes add water and wash into a 100 c.c. flask. (If a check analysis as suggested was made, wash this into a 50 c.c. flask.) Dilute to the mark and place 10 c.c. (or the equivalent of 1 tablet) in a 100 c.c. flask, add about 50 c.c. water and a few drops more KOH solution (20 per cent.) than is required to neutralize the acid. (Do not use NaOH.) Dilute to the mark and compare the colour with that produced by a standard nitrate solution similarly treated. Use any convenient colorimeter or Nessler tubes. Reagents and Standards: *Phenoldisulphonic Acid Reagent.*—Dissolve 25 Gm. of pure white phenol in 150 c.c. of concentrated  $\text{H}_2\text{SO}_4$ , add 75 c.c. of fuming  $\text{H}_2\text{SO}_4$  (13 per cent.  $\text{SO}_3$ ), stir well and heat for 2 hours at about  $100^\circ\text{C}$ . *Standard Solution.*—Dissolve 0.7217 Gm. pure  $\text{KNO}_3$  in 1 litre of water. Evaporate 10 c.c. of this solution just to dryness on the steam-bath. Cool and treat the residue with 2 c.c. phenoldisulphonic acid reagent, observing the precautions noted above and using a glass rod if necessary to aid the solution of the residue. After 5 or 10 minutes dilute to 250 c.c. Each c.c. of this solution contains 0.004 Mgm. nitrogen. Add an excess of KOH solution to an aliquot of this solution and dilute to 100 c.c. It is advisable to prepare a standard of approximately the same colour

as the unknown. Nitroglycerin is 5.4 times nitrate nitrogen. *Estimation by the Modified Hay Method.*—Of the above described  $\text{Et}_2\text{O}$  extract place 10 c.c. in 120 c.c. Erlenmeyer flask, dilute with 5 or 10 c.c. of  $\text{EtOH}$  and add about 5 c.c. of 0.5 per cent. alcoholic  $\text{KOH}$ . Cover with a watch glass and allow to stand 10 minutes. Place on steam-bath, allow to boil, remove the watch glass, and when most of the liquid is evaporated, add about 25 c.c. water and leave on steam-bath until the odour of  $\text{EtOH}$  can no longer be detected. Cool and dilute to 250 c.c. Each c.c. of this solution represents 0.01 of a tablet. Introduce 5 c.c. representing 0.0324 Mgm. nitroglycerin into a 100 c.c. graduated flask, dilute with sufficient water to make the volume 90 to 95 c.c., add one drop concentrated  $\text{HCl}$ , then 2 c.c. sulphanic acid solution and 2 c.c. naphthylamine hydrochloride solution. Complete the volume with water. Prepare at the same time and in the same way, standards containing known amounts of  $\text{NaNO}_2$ , by taking 80 c.c. of the standard solution of  $\text{NaNO}_2$ , and adding one drop of concentrated  $\text{HCl}$ , 2 c.c. sulphanic acid solution and 2 c.c. naphthylamine hydrochloride solution, and completing volume to 100 c.c. with water. Stopper the flask and mix well. Compare the colours after 30 minutes, each c.c. is equivalent to 0.00064 Mgm. nitroglycerin. Reagents and Standards: *Sulphanilic Acid Solution.*—Dissolve 1 Gm. in 100 c.c. of hot water. *Naphthylamine Hydrochloride Solution.*—Under a hood boil 0.5 Gm. of the salt with 100 c.c. of water for 10 minutes, keeping the volume constant. Filter and keep in a glass-stoppered bottle. *Standard Solution of Sodium Nitrite.*—To a cold solution of about 2 Gm. of  $\text{NaNO}_2$  or  $\text{KNO}_2$  in 50 c.c. of water, add a solution of  $\text{AgNO}_3$  as long as a precipitate appears. Decant the liquid and thoroughly wash the precipitate with cold water. Dissolve in boiling water. On cooling the  $\text{AgNO}_3$  is precipitated. Dry the crystals in the dark at the ordinary temperature (preferably in a vacuum). Weigh out 220 Mgm. of the dry  $\text{AgNO}_3$ , dissolve in hot water and decompose with a slight excess of  $\text{NaCl}$ . When the solution becomes clear, dilute to 1 litre. Dilute 5 c.c. of this solution to 1 litre. This second dilution is the standard to be used. It contains 0.0001 Mgm. nitrite per c.c. Only nitrite-free water should be used in the estimation by the Modified Hay method.

Of the above methods the Scoville is more generally employed because of the rapidity with which it can be operated.

It is to be remembered, however, that both of the above methods involved a colorimetric comparison, and that different operators are better able to judge one colour in preference to another. The Scoville method gives a yellow-coloured solution, while the Hay method yields a rose-coloured solution. So far as accuracy is concerned, apart from the end colour comparison neither method is to be preferred to the other.

**Nitrometer, Simple, for Nitrous Ether Assays.** T. J. Bradley. (*Drugg. Circ.*, 1914, 58, 708.) The instrument consists



of a glass tube about 16 inches long, and graduated, from the top downward, to 50 c.c. in fifths. At the top this graduated tube is contracted and has a stopcock connecting it with a cylindrical funnel, which is also graduated at 5 c.c. and 10 c.c. The graduated tube, below the graduation, is expanded to form a bulb of about 75 c.c. capacity, and below this there is a side tube with an open end to be connected with a levelling bulb. The bottom of the instrument is closed and it stands on a removable base, preferably of iron. The levelling bulb is connected with the side tube by about two feet of flexible rubber tubing and is supported by a clamp which is attached to the graduated tube and easily adjusted at any height.

**Parchment Paper, Tests for Distinguishing, from Pergamyn Paper.** G. Annoni and G. Rodano. (*Ann. Lab. Chim. delle Gabelle*, 1914, 7, 19; *J.S.C.I.*, 1915, 34, 487.) Parchment paper, prepared by the action of  $\text{H}_2\text{SO}_4$  on rag paper, is resistant to boiling water and to a boiling 2 per cent. solution of  $\text{K}_2\text{CO}_3$ ,

whereas the imitation pergamyn papers, prepared by mechanical treatment of wood pulp, are much less resistant to water and are immediately disintegrated by a boiling 2 per cent. solution of  $\text{K}_2\text{CO}_3$ . On treatment with a drop of  $\text{ZnCl}_2$ -I solution, a violet stain is produced on both kinds of paper, though more slowly on the pergamyn, but on subsequent treatment with water, the violet changes to an intense blue—due to hydrocellulose—in the case of parchment paper, whilst only a faint violet coloration is left on pergamyn paper. Pergamyn papers

invariably contain resin, whilst this is absent from parchment papers; hence the production of a reddish violet coloration on treatment with acetic anhydride and  $\text{H}_2\text{SO}_4$  in succession is a proof that the sample is a pergamin paper.

**Pearl Barley, Facing of.** J. F. Liversoege and H. Hawley. (*Journ. Soc. Chem. Ind.*, 1915, **34**, 203.) The total ash of unfaced pearl barley should not exceed 1.1 per cent. and the insoluble ash 0.1 per cent. In a large proportion of normal pearl barley the latter figure does not exceed 0.05 per cent. The authors employ 10 Gm. of material for an ash determination, and approximately N/HCl to remove the soluble constituents from the ash.

**Petrolatum Liquid, or Russian Mineral Oil.** (*Amer. J. Pharm.*, 1914, **86**, 322.) The following is an abstract of the report of the Council on Pharmacy and Chemistry submitted to the Amer. Pharm. Assoc. The following list of fancy trade names for the hydrocarbon is given:—Adepsine oil; amilee; atoleine; alolin; blandine; crysmalin; deeline; glyco; glycoline; glymol; heavy petroleum oil; liquid albolene; liquid cosmoline; liquid fossiline; liquid geoline; liquid paraffin; liquid petrolatum; liquid saxoline; liquid vaseline; mineral glycerin; mineral oil; neutralol; olo; paraffin oil; paroline; petro; petrolax; petrolia; petrolol; petronol; petrosio; rock oil; Russian liquid petrolatum; Russian mineral oil; Russian paraffin oil; russol; saxol; terralbolia; terraline; usoline; water-white mineral oil; white paraffin oil. Liquid petrolatum has been included in many pharmacopœias, under the following names: *Petrolatum liquidum*, U.S.P.; *Paraffinum liquidum*, pharmacopœias of Great Britain, Germany, the Netherlands, Japan, Belgium, Austria, Denmark, Switzerland, Sweden, Serbia, Italy, Hungary and Russia; *Oleum Paraffinae*, Spanish Pharmacopœia; *Vaselinum liquidum*, French Pharmacopœia, and *Oleum vaselini* (as a synonym), pharmacopœias of Denmark and Russia.

The following are the requirements for sp.g. of the different pharmacopœias at 15°C. unless otherwise stated; *U.S.P.* VIII, 1905, 0.870 to 0.940 at 25°; *Ph. Brit.* IV, 1898, 0.885 to 0.890 at 15.5°; *B.P.C.* II, 1911, usually 0.875 or lower; *Ph. Germ.* V, 1910, at least 0.885; *Ph. Russ.* VI, 1910, 0.880 to 0.885; *Ph. Hung.* III, 1909, 0.88 to 0.89; *Ph. Ital.* III, 1909, 0.875

to 0.890 ; *Ph. Fr.* V, 1908, about 0.875 ; *Ph. Serb.* II, 1908, about 0.880 ; *Ph. Svec.* IX, 1908, 0.88 to 0.90 ; *Ph. Helv.* IV, 1907, 0.880 to 0.885 ; *Ph. Dan.* VII, 1907, at least 0.880 ; *Ph. Austr.* VIII, 1906, at least 0.880 ; *Ph. Belg.* III, 1906, not below 0.880 ; *Ph. Jap.* III, 1906, 0.875 to 0.945 ; *Ph. Ned.* IV, 1905, not below 0.860 ; *Ph. Hisp.*, 1905, 0.840. For pharmaceutical purposes liquid petrolatum may be divided into two grades : a lighter and more limpid oil used extensively for oil sprays ; and a heavier, more viscous oil, as generally recognized in European pharmacopœias, used as an ointment vehicle, and also as a remedy for intestinal stasis. For the *U.S.P.* IX it is proposed to make the official requirements for a colourless or slightly transparent liquid free from fluorescence without odour or taste ; sp.g. from 0.845 to 0.940 at 25°C.

Since the definition of liquid petrolatum in the *U.S.P.* permits the use of fluorescent products of widely varying specific gravities, it is recommended that physicians who desire the water-white non-fluorescent (Russian) mineral oil should use the term "*Petrolatum Liquidum, Grave*," or "*Paraffinum Liquidum, B.P.*," if the heavy product is desired, and "*Petrolatum Liquidum, Leve*," if the light varieties are required. It is further recommended that under the foregoing names manufacturers and pharmacists be requested to dispense the products in accordance with the following descriptions : *Petrolatum Liquidum, Grave*.—Heavy (Russian) Liquid Petrolatum.—*Paraffinum Liquidum, B.P.* (1898), liquid paraffin.—A transparent, colourless, tasteless, non-fluorescent, oily liquid, odourless when cold but giving off a faint petroleum odour on heating. This preparation should correspond to the requirements of the *B.P.* (1898) for liquid paraffin and have a specific gravity of about 0.885 to 0.890 at 15°C. This is the type of preparation used for internal administration. It is also used as a basis for ointments and salves and as a local application to wounds, ulcers and in certain forms of skin diseases in which a simple protective is desired. *Petrolatum Liquidum, Leve*.—Light (Russian) Liquid Petrolatum.—A transparent, colourless, tasteless, non-fluorescent, oily liquid, odourless when cold, but giving off a faint petroleum odour on heating. In other respects this preparation should correspond to the pharmacopœial tests for liquid petrolatum and have a specific gravity of about 0.860 to 0.875 at 15°C. This is a type of preparation extensively used as a vehicle for the oily sprays in nose and throat work. It is

also being used as one of the constituents in the now popular paraffin oil cold cream and has been used to some extent for internal administration in the treatment of chronic stasis. Being more limpid than the heavier preparation it is more readily taken, though greater care must be exercised in securing a sample devoid of the lighter fractions of petroleum distillates.

**Phenolphthalein, New Method for Determining.** A. Mirkin. (*Amer. J. Pharm.*, 1914, 86, 307.) 1 Gm. of phenolphthalein, 0.8 Gm. of hydroxylamine hydrochloride, and 0.52 Gm. of 90 per cent. NaOH, finely powdered, are dissolved in 35 to 40 c.c. of absolute EtOH and boiled for 2 or 3 hours under a reflux condenser until the liquid turns yellow. The liquid is then diluted with water, transferred to a 250 c.c. volumetric flask, 10 c.c. of 10 per cent.  $H_2SO_4$  is added and the flask filled to the mark with water. Fifty c.c. is taken for titration. First the acid is neutralized, using methyl orange as indicator. Then the excess of hydroxylamine is titrated with N/10 KOH, using phenolphthalein as indicator. A blank is run, using the same amounts of hydroxylamine, NaOH and alcohol, and boiled for the same length of time. The difference in the number of c.c. of N/10 alkali used in the titration of the blank experiment and in the sample, multiplied by 316, gives the quantity of phenolphthalein. When applying the method to medicinal tablets, the tablets were placed in a cylinder and crushed under EtOH with a glass rod. The alcohol was decanted off through a filter into a volumetric flask and the extraction and decantation continued until complete extraction was obtained. An aliquot part of the extract was then taken for the determination. The method gives very accurate results. The yellow colour of the oxime does not interfere with the titration, as by proper dilution it colours the liquid only slightly. Tablets of phenolphthalein frequently contain milk sugar or cane sugar, but as cane sugar does not give an oxime with hydroxylamine, and as milk sugar is practically insoluble in absolute EtOH, they do not interfere with the reaction. In case, however, that the method should give too high a result, it is better to make a volumetric determination of sugar.

**Phenols and Phenolic Ethers with Unsaturated Side Chains, Behaviour of, towards Ozone.** C. Harries and R. Haarmann. (*Ber.*, 1915, 48, 32; *J.S.C.I.*, 1915, 34, 573.) The earlier results obtained in the formation and fission of the ozon-

ides of unsaturated phenols such as eugenol were not entirely satisfactory. The problem has therefore been re-examined in the case of the substances detailed below, of which it was found generally possible to prepare the ozonides by treatment of a solution with well-dried 1 per cent. ozone. In hexane solution isoeugenol gave a yellow, syrupy ozonide,  $C_{10}H_{12}O_5$ , which, when produced in acetic acid solution, underwent decomposition, giving acetaldehyde and a 25–38 per cent. yield of vanillin, together with resinous substances. It was found that mere treatment of an acetic acid solution of isoeugenol with a current of oxygen for 120 hours also effects partial oxidation to vanillin, this substance being produced to the extent of approximately 12 per cent. By reducing isoeugenol ozonide in  $Et_2O$  solution by means of zinc dust and acetic acid, vanillin could be obtained to the extent of 71 per cent. of the theoretical yield. Isoeugenyl acetate is remarkable in combining with more than the expected proportion of ozone, the ozonide produced in ethyl acetate solution being a crystalline solid of the composition  $C_{12}H_{14}O_9$ . On fission with acetic acid, this ozonide gives rise to acetyl-vanillic acid in poor yield. Eugenol ozonide, obtained by the action of 1 per cent. ozone on a solution of eugenol in ethyl acetate, is an oily substance which is considerably more explosive than isoeugenol ozonide. When decomposed by warming with acetic acid, no formation of homovanillin is observable, the only product being a brown oil, b.p.  $120^{\circ}$ – $200^{\circ}C.$  at 0.5 mm., which rapidly resinifies. Reduction of eugenol ozonide in ethereal solution by means of zinc dust and acetic acid caused fission with formation of homovanillin,  $CH_3O.C_6H_3(OH).CH_2.CHO$ , a colourless, viscous oil, b.p.  $110^{\circ}$ – $114^{\circ}C.$  at 0.45 mm., with a smell resembling vanilla; *p*-nitrophenylhydrazone, deep yellow needles, m.p.  $150^{\circ}$ ; semicarbazone, prisms, m.p.  $173^{\circ}C.$ ; oxime, leaflets or needles, m.p.  $115^{\circ}C.$ ; bisulphite compound, colourless powder. Eugenyl acetate, in contrast to isoeugenyl acetate, forms a normal ozonide,  $C_{12}H_{14}O_8$ , which is conveniently obtained by the action of 1 per cent. ozone on a solution in hexane; the ozonide separates from ethereal solution in colourless tablets or needles, m.p.  $63^{\circ}C.$  Fission of the ozonide by acetic acid produces acetylhomovanillic acid, acetylhomovanillin, and vanillin. Eugenol methyl ether ozonide, on fission by acetic acid, gave methylvanillin. Reduction of the ozonide yielded a yellow liquid, b.p.  $112^{\circ}$ – $113^{\circ}C.$  under 0.6 mm., of which a preliminary examination indicated it to

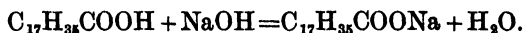
be methylhomovanillin (*p*-nitrophenylhydrazone, m.p. 157°; semicarbazone, m.p. 181°). The above results provide an explanation for the contradictory results of earlier investigators, who have found it possible to obtain vanillin from isoeugenol, using oxygen relatively poor in ozone, whilst later investigators with more effective ozone apparatus applied too concentrated ozone and obtained only resinous products.

**Salicylic Acid, New and Sensitive Reaction for.** P. A. W. Self. (*Pharm. J.*, 1915 [4], 40, 521.) The reagent is prepared by mixing equal parts by volume of 40 per cent. formaldehyde solution and concentrated  $H_2SO_4$  (it is important that these proportions should be adhered to), and the mixture is cooled thoroughly. Moisten the substance to be tested, in a porcelain dish, with the above mixture, add a little ammonium vanadate, and stir well. If salicylic acid is present, a Prussian blue colour appears immediately, varying in intensity with the amount of salicylic acid, and rapidly changes, first to a greenish blue and finally to green. If, however, no salicylic acid or other substance capable of yielding a colour reaction is present, the colour given by the reagents alone is a yellowish red or orange, which after 2 or 3 minutes begins to change to greenish yellow and finally becomes green. The quantities of reagents used should be adjusted roughly to the amount of substance to be tested; for a minute trace, not more than sufficient of the liquid to moisten it should be employed, together with a fraction of a milligramme of ammonium vanadate; while with about a milligramme of salicylic acid, two drops of the formaldehyde and sulphuric acid mixture, and about 2 or 3 Mgm. of ammonium vanadate are suitable quantities. When these precautions are taken 0.02 Mgm. of salicylic acid gives a very distinct colour, while with about 1 Mgm. the colour is intense. Acetylsalicylic acid and salicin do not give this reaction, nor do a number of other organic substances which are enumerated.

**Soap, Determination of Free Alkali in.** W. Huggenberg. (*Zeits. angewandte Chem.*, 1914 [4]; *Schwei. Apoth. Zeit.*, 1914, 52, 598.) Five Gm. of the soap is dissolved in 100 c.c. of neutral 50 per cent. EtOH, in a flask fitted with a tube condenser, by warming on the water-bath. After cooling the solution is treated with 15 to 20 c.c. of neutral solution of  $BaCl_2$  and, without filtering, titrated with N/40 stearic acid solution. This



is prepared by dissolving 7.1 Gm. of stearic acid in 1 litre of absolute EtOH. It is standardized against N/10 NaOH a 1 : 4000 solution of  $\alpha$ -naphtholphthalein as indicator. The same indicator is used in the soap titrations. The equivalent of NaOH for the amount of N/40 stearic acid used is found from the equation



If the amount of free alkali as  $Na_2CO_3$  is required another titration is performed before adding  $BaCl_2$ , which gives the total free alkali. The difference between the two titrations gives the equivalent of the  $Na_2CO_3$  present. For textile processes [and especially silk industries] the soap should be free from NaOH; the extreme limit must not exceed 0.08 per cent.

**Soap, Qualitative Test for Silicates in.** H. W. Leitch. (*J. Ind. Eng. Chem.*, 1914, 6, 811.) Dissolve about 1 Gm. of the soap in 25 c.c. of water, and add N/HCl 5 c.c. in excess of the amount necessary to neutralize the total alkali, using methyl orange indicator. Heat on the water-bath until the fatty acids float to the top and the liquid beneath is clear. Filter through ordinary filter paper, make the filtrate neutral or slightly alkaline with N/NaOH, add 10 c.c. of this solution corresponding to about 0.3 Gm. of the original sample, to 5 c.c. of normal alcoholic caustic potash, and boil down to 10 c.c. on the steam-bath. Pour this filtered solution into a test tube containing 10 c.c. of acetone and 1 c.c. of a solution made by dissolving 10 Gm. of pure sodium aluminate and 2 Gm. of NaCl in a litre of water. If water glass is present in the sample of soap, a flocculent gelatinous precipitate results. Dextrin or starch, if present in the soap, will also precipitate here, so that if a test made by adding a drop of I solution to a portion of the filtrate from the acidified soap solution shows a blue or reddish brown coloration, a change in the procedure must be made just before the boiling with alcoholic KOH.

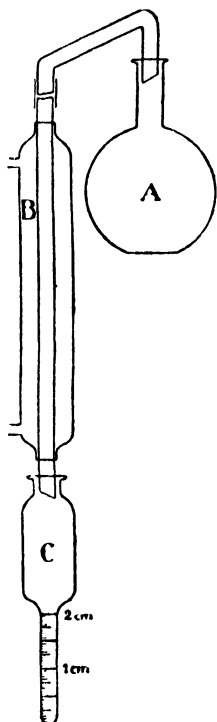
**Sodium Glycerophosphate, Constitution of Crystallized.** L. Grimbert and O. Bailly. (*Comptes rend.*, 1915, 160, 207-10.) Na glycerophosphate, prepared by Poulenc's method, is a mixture of two salts, a crystalline salt and an amorphous compound. In order to remove the last traces of glycerol, the mixed salts were converted into the Ca salts.

When treated at room temperature with 10 c.c. Br water, 0.25 Gm. of the Ca salt derived from the former gave no coloration with resorcinol and  $\text{H}_2\text{SO}_4$ , and no colour test when treated with a mixture of 4 per cent. aqueous KI and 5 per cent. alcoholic  $\text{o-HOC}_6\text{H}_4\text{CO}_2\text{H}$  with the subsequent addition of  $\text{H}_2\text{SO}_4$ . The Ca salt derived from the amorphous glycerophosphate, however, after oxidation with Br, gave a brilliant red colour with the resorcinol mixture and a violet colour with the KI- $\text{HOC}_6\text{H}_4\text{CO}_2\text{H}$  mixture, indicating the presence of a ketonic group. The oxidation product of the amorphous salt also forms an osazone and reduces Fehling and Nessler solutions in the cold. The authors therefore conclude that the crystalline salt is  $\beta$ -glycerophosphate,  $\text{O} : \text{P}(\text{ONa})_2 \cdot \text{O} \cdot \text{CH}(\text{CH}_2\text{OH})_2 \cdot 5\text{H}_2\text{O}$ , and the amorphous salt the  $\alpha$ -glycerophosphate,  $\text{O} : \text{P}(\text{ONa})\text{O} \cdot \text{CH}_2 \cdot \text{CH}(\text{OH}) \cdot \text{CH}_2\text{OH}$ , which on oxidation passes into  $\text{O} : \text{P}(\text{ONa})_2 \cdot \text{O} \cdot \text{CH}_2\text{COCH}_2\text{OH}$ .

**Spices, Chemical Analysis of. C. Arragon.** (*Schweiz. Apoth. Zeit.*, 1915, 53, 220.)

**Determination of Water.**—Ten Gm. of the material is introduced into the flask A with 60 c.c. of pure, previously dried oil of turpentine. Distillation is then performed rapidly until the receiver C, which has a total capacity of 50 c.c., is practically full. This is then detached, and centrifugated for 15 minutes. The tube is then placed in a water-bath at  $15^\circ\text{C}$ . and the volume of water which has separated is read off. Each graduation is equivalent to 0.5 per cent. of water. It is applicable to other powders, such as flour, which do not contain volatile constituents besides water.

**Determination of Fat and Essential Oil.**—Ten Gm. of the material is exposed for 6 hours in a desiccator over  $\text{H}_2\text{SO}_4$ . It is then extracted for 8 hours with dry  $\text{Et}_2\text{O}$ . The solvent is distilled off from a tared flask slowly, at the rate of 1 drop a second. When distil-



lation ceases, the last traces of  $\text{Et}_2\text{O}$  are blown off in a water-bath, for two minutes, with a current of air. The residual fat and essential oil is weighed. The flask is then fitted with a cork bearing two tubes. One of these is drawn out to a fine point, which impinges on the surface of the residue. The other is connected up with a condenser. A current of steam is then passed through the drawn out tube and condensed in the usual manner, through the other tube. When water accumulates in the flask, it is distilled over by means of a small flame. Distillation is continued until all essential oil has been removed, and the aqueous distillate measures about 250 c.c. The residue in the flask is then shaken out with successive quantities of  $\text{Et}_2\text{O}$  until all soluble matter is removed. The bulked  $\text{Et}_2\text{O}$  solution is evaporated, and the residue dried and weighed. This weight of fatty matter, deducted from the weight of the total  $\text{Et}_2\text{O}$  extract previously determined, gives the amount of essential oil.

*Determination of Starch.*—Two Gm. of the material is boiled under a tube condenser with 150 c.c. of water, and 15 c.c. of  $\text{HCl}$ , sp.g. 1.125, for 90 minutes. After cooling and neutralizing with solution of  $\text{NaOH}$ , 1 : 10, 5 c.c. of basic lead acetate is added. The volume of liquid is then adjusted to exactly 250 c.c., and filtered. Twenty-five c.c. of clear filtrate is then taken for sugar determination with Fehling's solution in the usual manner. The percentage of dextrose found, multiplied by 0.9, gives the percentage of starch present

*Cellulose* may be determined by Koenig's method.

A table of results with over thirty samples is given. [In this the figures for "essential oil" in some instances differ from those usually obtained from certain spices in this country.—Ed. Y.B.]

**Starch, Soluble, for Indicator, Preparation of.** R. M. Chapin. (*J. Ind. Eng. Chem.*, 6, 649.) Potato starch is digested 1 to  $1\frac{1}{2}$  hours with dilute  $\text{HCl}$ , on a boiling water-bath. The solution is made alkaline with  $\text{NH}_4\text{OH}$ ; about one-third its volume of 95 per cent.  $\text{EtOH}$  is added, and the solution filtered through muslin. It is then run at 40–45°C. in fine streams, into a large volume of 95 per cent.  $\text{EtOH}$  with stirring. The precipitated starch is filtered and washed with  $\text{EtOH}$  and dried. It is readily soluble in cold water, giving a good reaction with I, but is not quite so sensitive as ordinary fresh starch solution.

**Toxicological Material, Freezing to Aid Disintegration.** A. Le Roy. (*Comptes rend.*, 1915, 160, 313.) Putrid viscera and other soft material may be easily disintegrated if it is frozen hard before being passed through a mincing machine. It should be exposed to a temperature of  $-6^{\circ}$  to  $-10^{\circ}\text{C}$ . for 14 to 24 hours, in a laboratory refrigerator, or if this is not available, in an ice safe such as is used in all large towns, for cold storing perishable food. Failing these freezing mixtures may be employed. The frozen material is easily reduced to a fine state of division, and any putrid odour is materially lessened.

**Vanilla Extract, Acidity and Ash Value of.** A. L. Winton, A. R. Allright, and E. H. Berry. (*J. Ind. Eng. Chem.*, 1915, 7, 516.) It is suggested that the determination of the total acidity and ash values of vanilla extracts may serve to differentiate genuine from fictitious preparations. The following ranges in acidity and in ash values were found in 77 U.S.P. extracts made in the laboratory from different varieties, grades and lengths of vanilla beans. Total acidity, 30 to 52 c.c. N/10 alkali per 100 c.c.; acidity other than vanillin, 14 to 42 c.c. N/10 alkali per 100 c.c.; total ash, 0.220 to 0.432 Gm. per 100 c.c.; soluble ash, 0.179 to 0.357 Gm. per 100 c.c.; alkalinity of total ash, 30 to 54 c.c. N/10 acid per 100 c.c.; alkalinity of soluble ash, 22 to 40 c.c. N/10 acid per 100 c.c.

Practically the same values were obtained with and without the use of sugar or glycerin in the menstruum. Diminishing the strength of alcohol in the menstruum tended to increase the ash values and diminish the acidity. It is found that the acidity of commercial vanillin, m.p.  $78^{\circ}$  to  $78.4^{\circ}$  is slightly lower than the theoretical figure, being equivalent to 63 c.c. of N/10 alkali for 1 Gm. instead of 65.8 c.c. The possibility of developing a method of determining vanillin based on the acidity is suggested.

**Veronal, New Tests for.** H. Lucas. (*Pharm. J.*, [4] 1914, 39, 424.) Veronal, diethyl-barbituric acid, or diethyl-malonyl-urea, when fused with NaOH, at first gives an odour resembling that of crude acetone, and afterwards, fumes of  $\text{NH}_3$ . When boiled with aqueous NaOH and cooled, and then warmed again with solution of I, the odour of  $\text{CHI}_3$  is produced. Fused with solid NaOH and the residue treated with a drop of solution of  $\text{CuSO}_4$  it gives a purplish violet colour, due to the formation of copper biuret. A portion of the fusion mass dissolved in a little

water and introduced into a bottle with strong NaBrO solution gives a considerable volume of N, derived from the urea formed by the decomposition of the veronal.

**Wijs' Iodine Solution, Preparation of.** H. Dubovitz. (*Chem. Zeit.*, 1914, **38**, 1111; *J.S.C.I.*, 1915, **34**, 305.) To prepare Wijs' N/5 iodine monochloride solution, 7.8 Gm. of iodine trichloride and 8.5 Gm. of iodine are required per litre. Most text books, copying an error in a standard work, prescribe 9.4 Gm. of iodine trichloride and 7.2 Gm. of iodine per litre; the solution thus prepared is much less stable than that containing the correct amounts.

## PLANT ANALYSIS

**Acokanthera venenata from the Transvaal, Investigation of** (*Bull. Imp. Inst.*, 1915, **13**, 53.) The plants of the genus are well known arrow poisons, such as *Acokanthera ouabaio*, *A. deflersii*, *A. schimperi*, and *A. abyssinica*. From these two glucosides have been isolated, one crystalline, acokantherin ouabain, and another amorphous, variously known as abyssinin, amorphous acokanthein, amorphous ouabain or acokanthin. *A. venenata* was found to contain no alkaloid nor cyanogenetic glucoside; a bitter, intensely poisonous amorphous substance was isolated which had all the characters of Faust's crude acokantherin. The amount available was too small for treatment to isolate the crystalline glucoside. Physiological investigation of the bitter substance shows that it is a powerful heart poison, resembling digitalis in its action. In cases of poisoning chemical tests would be of little service in attempts to detect the toxic substance in consequence of its ill-defined nature and the extremely minute quantity necessary to produce a lethal result. (See also *Y.B.*, 1913, 134, and *Gen. Index*.)

**Ambrosia artemisifolia, Bitter Principles of.** B. E. Nelson and G. W. Crawford. (*J. Amer. Chem. Soc.*, 1914, **36**, 2536.) The American ragweed, *Ambrosia artemisifolia*, has yielded the authors a white crystalline substance, and an amorphous bitter principle. The latter is probably identical with "absinthin." These were obtained from the alcoholic extract of the herb, which was freed from resinoids by water and  $\text{Al}_2(\text{OH})_6$  and  $\text{Pb}_2\text{C}_2\text{H}_3\text{O}_2$ . The aqueous liquid was then shaken out with  $\text{Et}_2\text{O}$  until no longer bitter. The residual  $\text{Et}_2\text{O}$  extract when purified with  $\text{EtOH}$

and animal charcoal gave a crop of white prismatic tasteless crystals, m.p.  $208^{\circ}\text{C}.$ ; apparently inert physiologically, except for a slight sternutatory action when inhaled. These crystals were embedded in a yellow amorphous substance. The latter was removed by washing with  $\text{Et}_2\text{O}$  in which it was much more soluble. After repeated purifications this proved to be the bitter principle. It became indefinitely crystalline after prolonged standing over  $\text{H}_2\text{SO}_4$ . With strong  $\text{H}_2\text{SO}_4$  it affords a brown colour passing to purple, and with Froehde's reagent a green tint. After hydrolysis, it slowly reduces Fehling's reagent.

**Andrographis paniculata, Constituents of.** K. B h a d u r i. (*J. Amer. Pharm. Assoc.*, 1914, **86**, 349.) From the alcoholic extract of the herb two bitter principles have been extracted. One was crystalline pale yellow, m.p.  $206^{\circ}$ . It gave off a fragrant odour when heated. It was neither alkaloidal nor glucosidal in character. The empirical formula was  $\text{C}_{19}\text{H}_{28}\text{O}_5$ ; soluble in water. The second substance had an intensely bitter taste. It was a white amorphous powder, almost insoluble in water; m.p.  $185^{\circ}$ ; empirical formula  $\text{C}_{19}\text{H}_{31}\text{O}_5$ . It is proposed to name this "*Kalmeghin*." This was treated with an acid, and a white insoluble acid substance was formed,  $\text{C}_{14}\text{H}_{23}\text{O}_2$ ; for this the name kalmeghic acid is proposed.

**Anthemis nobilis, Constituents of the Flowers of.** F. B. P o w e r and H. B r o w n i n g, Junr. (*Proc. Chem. Soc.*, 1914, **30**, 210.) Belgian chamomile flowers were used. Percolation of 21.09 kg. with hot  $\text{EtOH}$  yielded 8.33 kg. of a viscid extract, which, on steam distillation, gave a volatile oil which had been previously investigated. The aqueous liquid (1) separated fatty matter and resin (2). (1): On standing, a small amount of apigenin glucoside precipitated. Concentration of the filtrate and extraction with  $\text{Et}_2\text{O}$  gave a bitter mass, containing no alkaloids.  $(\text{NH}_4)_2\text{CO}_3$  extract: Only 3,4-(HO) $_2\text{C}_6\text{H}_3\text{CH}:\text{CHCO}_2\text{H}$  was isolated. From the  $\text{Na}_2\text{CO}_3$  extract only a little apigenin could be obtained, while the  $\text{NaOH}$  extract, on acidification, gave a small amount of needles, m.p.  $143^{\circ}$ . The aqueous liquid extracted with  $\text{Et}_2\text{O}$  was then extracted a number of times with warm  $\text{AmOH}$ , from which, on concentration, was obtained a viscid product, from which, after removal of the solvent and repeated separations from dilute  $\text{EtOH}$ , 30 per cent.  $\text{AcOH}$ , and 20 per cent.  $\text{EtOH}$ , were obtained faintly yellow crystals of *apigenin-d-glucoside* (A),  $\text{C}_{21}\text{H}_{20}\text{O}_{10} \cdot 3\text{H}_2\text{O}$ ,  $1\text{H}_2\text{O}$  remaining above  $125^{\circ}$ , giving a purple-

brown colour with  $\text{Fe}_2\text{Cl}_6$ , m.p.  $178^\circ\text{--}180^\circ\text{C}$ . (decomp.), differs from Vongerichten's compound by  $1\text{H}_2\text{O}$  and its stability on boiling in water. The aqueous liquid, after the extractions with  $\text{Et}_2\text{O}$  and  $\text{AmOH}$ , was treated with basic lead acetate (nothing isolated from the precipitate), and the filtrate, after removal of the Pb, concentrated to a syrup. From this were finally obtained  $\alpha$ -phenylglucosazone, showing the presence of a hexose, choline (isolated as the chloride and converted into the Au and Pt double salts), and *dl*-inositol, separated as the hexa-acetate. (2): Extraction with acid gave only a small amount of choline. *Petroleum ether extract*:  $\text{C}_{30}\text{H}_{62}$ ; taraxasterol,  $\text{C}_{29}\text{H}_{47}\text{OH}$ ; the acetate has  $[\alpha]_D$   $102.5^\circ$ , not  $122.2^\circ$ ; a glucoside could not be prepared. Oleic, linoleic, and cerotic acids, and what was probably a mixture of stearic and palmitic acids. *Et<sub>2</sub>O extract*: Gradually deposited a phytosterolin (phytosterol glucoside) which contained chiefly sitosterol and less stigmasterol. In small amounts, *p*- $\text{HOC}_6\text{H}_4\text{Ac}$ , apigenin, and *p*- $\text{HOC}_6\text{H}_4\text{CO}_2\text{H}$ . "Anthemic acid" and Klobb's "anthesterol" appear to have been indefinite products. The bitter taste of chamomile flowers seems to be due to dark-coloured amorphous substances.

**Arabia sinensis, Chemical Constituents of the Bark of.** H. Kondo and J. Okada. (*J. Pharm. Soc. Japan*, 1914 [394], 1366; *Chem. Abstr.*, 1915, 9, 1092.) This bark is used by Japanese as a remedy for diabetes. The powdered bark was extracted with petroleum ether,  $\text{Et}_2\text{O}$ ,  $\text{EtOH}$ , and boiling water in the order named. The petroleum ether contained fats, chlorophyll, etc., and was not further examined. In the  $\text{Et}_2\text{O}$  extract protocatechuic acid was found, and also a yellow amorphous substance which yielded protocatechuic acid when heated with alcoholic KOH. The  $\text{EtOH}$  extract contained amorphous glucosides precipitable by lead acetate; soluble only in water or  $\text{EtOH}$ ; these yielded sapogenin. Choline was found in the  $\text{EtOH}$  extract not precipitable by lead acetate. The aqueous extract yielded mucic acid when precipitated by  $\text{EtOH}$  and oxidized with  $\text{HNO}_3$ .

**Brehmia Spinosa Seeds, Investigation of.** (*Bull. Imp. Inst.*, 1915, 13, 52.) This Loganiaceous plant, also known as *Strychnos spinosa*, Lam., is known in Seychelles as "Calebassia." Examination of the seeds confirm the statement of Flueckiger that they contain no strychnine, nor brucine, nor other alkaloid.

**Clematis vitalba, Constituents of.** F. Tutin and H. W. B. Clewer. *Proc. Chem. Soc.*, 30, 210.) No alkaloid could be found, and the statements regarding the acrid, irritating properties of the plant could not be confirmed. The flowering branches were used, losing 64.9 per cent. in weight on drying. Only a trace of material volatile with steam was obtained. The EtOH extract was taken up with water: after passing steam through this the non-volatile residue gave an aqueous liquid and resin. (1) *The Et<sub>2</sub>O extract* contained 3,4-(HO)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>CH:CHCO<sub>2</sub>H. *Amyl alcohol extract*: A caulosapogenin glucoside, which could not be isolated; caulosapogenin, C<sub>42</sub>H<sub>66</sub>O<sub>6</sub> (A). In benzoylating this, in addition to the tetra-benzoyl derivative there were obtained difficultly soluble needles, m.p. 252°, yielding (A) on hydrolysis, but not agreeing with any formula based on C<sub>42</sub>H<sub>66</sub>O<sub>6</sub>. A *methyl ether*, in needles, m.p. 229°C, was also prepared, which differed in analytical figures from those obtained previously by Power and Salway; although the  $\alpha_p$  was identical. The ether gave an anomalous *benzoyl derivative*, needles, m.p. 187°–188°. An explanation for these results has not been found. Filtrate from Pb(OH)OAc precipitation: (A); dextrose; NH<sub>4</sub> salts. Resin (2), 2.29 per cent. of the plant used, dissolved in alcohol, mixed with sawdust, and dried. *Petroleum ether extract*: On adding Et<sub>2</sub>O and keeping, a solid was obtained, which yielded melissic acid and myricyl alcohol. Unsaponifiable: ceryl alcohol (separated as the hydrogen phthalate), C<sub>31</sub>H<sub>64</sub>, a phytosterol, probably a mixture of sitosterol and stigmasterol. Acids; purified through the Me esters. The following fatty acids were purified as methyl esters: Palmitic, stearic acids, an isobehenic acid, m.p. 69.5°, cerotic acid; a mixture of unsaturated acids, chiefly linoleic acid. The Et<sub>2</sub>O extract contained a phytosterolin, m.p. 295°, giving an acetate with the formula C<sub>36</sub>H<sub>56</sub>O<sub>6</sub>(C<sub>2</sub>H<sub>3</sub>O<sub>2</sub>)<sub>4</sub> and myricyl alcohol; a crystalline saponin, C<sub>54</sub>H<sub>36</sub>O<sub>16</sub>.5.5H<sub>2</sub>O, m.p. 235°–240°C., yielding caulosapogenin on hydrolysis, but not identical with caulosaponin.

**Cotton-Root Bark, Chemical Examination of.** F. B. Power and H. Browning, Junr. (*Pharm. J.*, 1914 [4], 39, 420.) Cotton-root bark has a reputation as an emmeagogue and for arresting hæmorrhage. The results of its chemical investigation may be summarized briefly as follows:—On distilling an alcoholic extract of the bark in a current of steam a very small amount



of a pale yellow essential oil was obtained. This gave the colour reaction of furfuraldehyde, and on keeping it deposited a few crystals melting at  $112^{\circ}$ – $114^{\circ}$ , which appeared to consist of acetovanillone. The other constituents of the bark were found to comprise : (1) A phenolic acid, m.p.  $196^{\circ}$ – $199^{\circ}\text{C}$ ., which gives an intense blue colour with  $\text{Fe}_2\text{Cl}_6$ , and is probably 2 : 3-dihydroxybenzoic acid ; (2) salicylic acid ; (3) a new, colourless phenolic substance, m.p.  $258^{\circ}$ – $260^{\circ}\text{C}$ ., to which the formula  $\text{C}_9\text{H}_{10}\text{O}_8$  has been assigned, and which yielded an acetyl derivative melting at  $152^{\circ}\text{C}$ . It dissolves in aqueous alkalis, forming bright yellow liquids, and with  $\text{Fe}_2\text{Cl}_6$  a purple colour is produced. (4) A new, yellow phenolic substance, m.p.  $210^{\circ}$ – $212^{\circ}\text{C}$ ., which appears to possess the formula  $\text{C}_{13}\text{H}_{18}\text{O}_5$ , and which yielded an acetyl derivative, m.p.  $147^{\circ}$ – $149^{\circ}\text{C}$ . With aqueous alkalis and with concentrated  $\text{H}_2\text{SO}_4$  it gives a deep purple colour, and with  $\text{Fe}_2\text{Cl}_6$  a brown coloration is produced. (5) Betaine,  $\text{C}_5\text{H}_{11}\text{O}_2\text{N}$  ; (6) a fatty alcohol,  $\text{C}_{20}\text{H}_{42}\text{O}$ , m.p.  $77.5^{\circ}$ – $78.5^{\circ}\text{C}$ . ; (7) a phytosterol,  $\text{C}_{27}\text{H}_{46}\text{O}$ , m.p.  $130^{\circ}\text{C}$ . ; (8) a small amount of a hydrocarbon, m.p.  $60^{\circ}$ – $61^{\circ}\text{C}$ ., which apparently is triacontane,  $\text{C}_{30}\text{H}_{62}$  ; (9) ceryl alcohol,  $\text{C}_{27}\text{H}_{56}\text{O}$  ; (10) a mixture of fatty acids, consisting chiefly of oleic and palmitic acids. The bark contains, furthermore, a considerable proportion of sugar, from which *d*-phenylglucosazone, m.p.  $212^{\circ}$ – $214^{\circ}\text{C}$ ., was prepared, and by the acetylation of the sugar a small amount of penta-acetyl-dextrose was obtained. No alkaloid is contained in the bark, and no evidence could be obtained of the presence of tannin. The resinous material, from which some of the above-mentioned substances were isolated, was of a deep purplish colour, and amounted to 10.6 per cent. of the weight of air-dried bark employed.

**Cotton Seed, Poisonous Constituent of.** W. A. Withers and F. E. Carruth. (*Science*, 1915, 41, 324 ; *Chem. Abstr.*, 1915, 9, 1516.) The authors have separated from cotton-seed kernels a toxic substance which appears to be identical with a substance which Marchlewski isolated from crude cotton-seed oil and called gossypol. This substance is quickly oxidized in alcoholic  $\text{NaOH}$ . Its oxidation renders it non-toxic, and thus diminishes if it does not entirely remove the toxic properties of cotton-seed meal.

**Datura stramonium, Analysis of Leaves of.** L. E. Sayre and H. V. Cadwell. (*J. Amer. Pharm. Assoc.*, 1915, 4, 602.) The amount of total extractives obtained by treating

the drug successively with the following solvents together with the moisture and fibre is given as follows:—Moisture, 7.37;  $\text{CHCl}_3$  soluble, 9.71; soluble in 80 per cent. alcohol, 17.29; soluble in water, 9.37; soluble in dilute acid, 34.24; soluble in dilute alkali, 15.84; crude fibre extract, 0.54; cellulose, 4.64; residual ash, 1 per cent. The total alkaloid was found to be 0.28 per cent.

**Daviesia latifolia, New Constituent in.** Frank Tutin. (*J. Chem. Soc.*) Power and Salway (*Y.B.*, 1914, 144) described a new, crystalline, bitter substance, dibenzoylglucoxylose, as occurring in this plant. The author now finds that this substance is associated with a small proportion of a more sparingly soluble isomeride, isodibenzoylglucoxylose,  $\text{C}_{25}\text{H}_{28}\text{O}_{12}$ , crystallizing in small, bitter, colourless needles, m.p.  $173^\circ\text{--}174^\circ\text{C}$ . The occurrence of rutin in *Daviesia* leaves is confirmed.

**Digitalis, Ash Content of.** E. L. Newcomb and M. H. Haynes. (*Amer. J. Pharm.*, 1915, 87, 112.) From the examination of a number of authentic specimens and of commercial samples of good quality it is found that a pharmacopœial standard reading "Ash not to exceed 10 per cent." is too high and would exclude the genuine drug of good quality. The figures obtained by the author ranged from 6.61 to 14.4 per cent. of ash.

**Echinacea Roots, Examination of Two Species of.** F. W. Heyl and J. F. Staley. (*Amer. J. Pharm.*, 1914, 86, 450.) Two species of *Brauneria* furnish the Echinacea of American commerce, *B. angustifolia* and *B. purpurea*. Under the description of *Echinacea angustifolia* in the U.S. Dispensatory there is a confusion of two species. The bulk of the Echinacea root met with is derived from *Brauneria angustifolia*, the narrow-leaved purple cone flower. The residue is obtained from *B. purpurea*. The *B. angustifolia* roots investigated were collected in Kansas; neither they nor the roots of *B. purpurea* gave indication of the presence of any alkaloid. Both roots contained inulin and inuloid, cane sugar and reducing sugars and resins. Such medicinal activity as the drug may possess is attributed to these. The resins of *B. angustifolia* give indication of containing a crystalline substance; sufficient material from this species being available, they are being investigated further. The fresh root of *B. angustifolia* yielded 0.04 per cent. of amber-coloured essential oil.

**Fagara xanthoxyloides, A New Phytosterol from the Root Bark of.** G. T. Oestling. (*Ber. Pharm.*, 24, 308; *Chem. Abstr.* 1915, 9, 353.) *Fagara-phytosterol*,  $C_{27}H_{44}O$ , forms brilliant needles from EtOH, m.p.  $214^{\circ}C.$ ,  $[\alpha]_D +20.41^{\circ}$ . Its  $CHCl_3$  solution becomes faintly yellow with concentrated  $H_2SO_4$ , changing to faint red after 30 minutes; it adds Br in  $CHCl_3$  or EtOH solution. The acetyl derivative m.p.  $118^{\circ}C.$ , silky needles, adds Br.

**Flavone in the Farina or "Meal" of Primulaceous Plants.** H. Mueller. (*J. Chem. Soc.*, 1915, 107, 872.) The familiar dusty or mealy secretion found on the leaves, peduncles and fruits of different species of the N.O. Primulacae, and known by gardeners as "meal" or "farina," has been identified as flavone. The Chinese and Japanese primulas are notable for the quantity of this substance they secrete. It was possible to brush off some of this from plants of *Primula pulverulenta* and also by sifting the fruits. Some was also obtained from *P. japonica*. A further portion was removed by dissolving in  $C_6H_6$ ; but this was difficult to purify. The identity of the substance with flavone,  $C_{15}H_{10}O_2$  m.p.  $99^{\circ}-100^{\circ}C.$ , was established.

**Gentiana scabra, Japanese, Constituents of.** Y. Asahina and S. Yoda. (*Yakugakuzasshi*; *J. Pharm. Chim.*, 1914, 10, 376.) The authors at first named the sugar they have isolated from Japanese Gentian root "isogentianose" since it had a higher m.p.  $223^{\circ}C.$  instead of  $210^{\circ}C.$  attributed by Tanret to gentianose. They still find that this is the correct m.p. for the trisaccharide; but since its  $\alpha_D$  is identical with that of Tanret's gentianose, they admit that the two sugars are the same and abandon the distinctive name. The glucoside which they formerly obtained only in the amorphous state has since been crystallized and its identity with gentiopicroin established.

**Gloriosa superba Bulbs, Colchicine in.** H. W. B. Clewer, S. I. Green and F. Tutin. (*J. Chem. Soc.*, 1915, 107, 835.) Except for the note by Warden (*Y.B.*, 1881, 182), who found the plant to contain a bitter principle, "superbine," which was considered to be either identical with or closely allied to the bitter principle of squill, nothing has been published on the constituents of this poisonous Indian plant. The present investigation has shown that the bitterness and toxicity of the plant are due to the alkaloid colchicine, which has hitherto been known

to occur only in *Colchicum autumnale*. In the course of the investigation a number of other constituents of the plant were isolated—very small quantities of two other crystalline alkaloids ; choline ; dextrose ; a hydrocarbon ; a fatty alcohol ; a mixture of phytosterols containing stigmasterol ; a mixture of phytosterol glucosides including stigmasterol glucoside ; palmitic acid ; a mixture of unsaturated fatty acids ; benzoic acid ; salicylic acid ; and a hitherto unknown methoxy-salicylic acid—namely, 6-methoxy-2-hydroxybenzoic acid.

**Haricot Beans, HCN from.** H. Blair. (*Pharm. J.*, 1915 [4], 40, 586.) The generation of HCN from a sample of haricot beans, *Phaseolus lunatus*, is noted ; the beans purchased in the usual manner for culinary purposes. The HCN was generated from 1.00 Gm. of ground beans by maceration in water at 45°C., the acid washed out by passing a current of pure hydrogen at the rate of 10 litres per hour, and absorbing in dilute KOH solution. The amounts in the several time fractions are given herewith :—

Time in minutes.	HCN in Fractions of a
	Milligramme.
27 mins. . . . .	0.025 Mg.
49 „ . . . . .	0.050 „
87 „ . . . . .	0.080 „
117 „ . . . . .	0.100 „
198 „ . . . . .	0.120 „
358 „ . . . . .	0.160 „

[The occurrence of a cyanogenetic glucoside in some varieties of *Phaseolus lunatus* was first recorded by Dunstan and Henry (*Y.B.*, 1904, 140), and has been confirmed subsequently by many workers. The discoverers named the glucoside phaseolunatin. Jorissen (*Y.B.*, 1909, 49) pointed out the identity of this with linamarin, which was isolated by him from flax seed in 1891.—Ed. *Y.B.*]

**Hops, Nitrogenous Constituents of.** A. C. Chapman. (*Proc Chem. Soc.*, 1914, 30, 196.) In a number of samples of hops analyzed, the total percentage of nitrogen varied from 1.7 to 4 per cent., the percentages of soluble nitrogen varying from 0.44 to 0.9 per cent. The nitrogenous substances soluble in hot water consisted of soluble proteins, albumoses, ammonium salts, amino-compounds and amides, bases precipitable by phosphotungstic acid, and unclassified nitrogenous substances not precipitated by that reagent. Results were given showing the proportions in which these various classes of compounds

occur. In the course of the work, large quantities of hops and hop extract were worked with, and four distinct methods for the separation and isolation of the nitrogenous substances were employed. The following substances were isolated and identified: *l*-Asparagine, aspartic acid, betaine, choline, histidine, hypoxanthine, and adenine. Of these asparagine and choline have been previously detected. Two other bases were obtained, but in quantities too small to permit of their complete identification. One of these, however, was almost certainly arginine. Although considerable quantities of hops of various growths were employed, in no case was morphine, nor any alkaloid closely resembling it, obtained.  $\text{KNO}_3$  was present in appreciable quantities.

**Hornbeam Leaves, Volatile Constituents of.** T. Curtius and H. Franzen. (*Annalen*, 1914, 404, 93; *J.S.C.I.*, 1914, 33, 1020.) 1,500 kilos. of hornbeam leaves were distilled in steam in portions of 15 kilos., the distillate made alkaline with baryta water and redistilled to remove barium salts of volatile acids. The distillate was then treated with freshly precipitated  $\text{Ag}_2\text{O}$ , and the Ag salts of the acids thus produced from the aldehydes present were subsequently converted into the barium salts and separated from unchanged alcohols and ketones by again distilling. The following substances were isolated and identified: formic, acetic and hexylenic acids with one or more higher homologues of the latter, acetaldehyde, *n*-butylaldehyde, valerylaldehyde,  $\alpha\beta$ -hexylenealdehyde with several higher homologues, butylene, pentylene and hexylenic alcohols, and an alcohol of the formula,  $\text{C}_8\text{H}_{14}\text{O}$ , with one or more higher alcohols. The method previously used by the authors to detect the presence of formaldehyde in hornbeam leaves, oxidation of the volatile aldehydes with moist silver oxide and subsequent identification of formic acid, has been proved to be fallacious, as methyl alcohol is also oxidized to formic acid under similar conditions.

**Hydrocyanic Acid in Feeding-stuffs, Determination of, and its Occurrence in Millet and Guinea Corn.** J. R. Furlong. (*Analyst*, 1914, 39, 430.) One hundred Gm. of the ground material is extracted with 90 per cent. EtOH for 3 hours in a Soxhlet, and the extract, after expelling the EtOH, is distilled with 150 c.c. of 10 per cent.  $\text{H}_2\text{SO}_4$ , the distillate being collected in 5 c.c. of 10 per cent. KOH solution. At the end of 1 hour, the receiver is changed, water is added to the distillation flask, and

distillation continued. These operations are repeated until evolution of HCN ceases. The distillate is concentrated to 15 c.c., boiled for 10 minutes, after the addition of 1 c.c. of a 20 per cent. solution of  $\text{Fe}_2\text{SO}_4$  containing also 1 per cent. of  $\text{Fe}_2\text{Cl}_6$ , cooled, acidified with HCl, and 10 c.c. of glycerin added. After standing for about 18 hours, the mixture is transferred to a graduated cylinder, diluted to 50 c.c. with water, and the blue coloration compared in tubes of 1 in. diameter with standards prepared from known quantities of HCN. When the amount of HCN present is not less than 0.001 Gm. the standards may be made up directly, but with smaller quantities it is necessary to dilute to 150 c.c. and then concentrate as in the preparation of the solution from the plant material. From determinations of HCN in millet and Guinea corn plants of various ages, it was found that all the young plants contained a cyanogenetic glucoside, whilst the full-grown plants were free from this substance. In the case of Guinea corn, the yield of HCN reached a maximum (0.01 per cent.) in the 12-inch plants, and decreased as growth proceeded. With millet, the maximum amount of HCN (0.045 per cent.) was found in the 24-inch plants.

**Khaya senegalensis Bark, Investigation of.** (*Bull. Imp. Inst.*, 1915, 13, 49.) No alkaloid, glucoside or crystalline substance was found in the bark. The bitter taste was due to a tannin, of which the bark contained 10.2 per cent. It has no value for medicine.

**Lycoperdon gemmatum, Phytosterol-like Substance from.** T. I k e g u c h i. (*Zeit. physiolog. Chem.*, 1914, 92, 257; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 473.) The  $\text{Et}_2\text{O}$  extract of the dried fungus yields to alcohol a crystalline phytosterol-like substance,  $(\text{C}_{10}\text{H}_{16}\text{O})\eta$ ; in needles, m.p.  $283^\circ\text{--}284^\circ\text{C}$ .  $[\alpha]_D - 65.2^\circ$ . It does not acetylate and gives no digitonin-compound. With Liebermann's test it gives a yellow colour, passing to dark green; a slight reaction is obtained with Sulkowski's test and a reddish violet colour with  $\text{HC}_2\text{H}_3\text{O}_2$  and  $\text{H}_2\text{SO}_4$ .

**Matricaria chamomilla, The Constituents of the Flowers of.** F. B. P o w e r and H. B r o w n i n g, Junr. (*Proc. Chem. Soc.*, 1914, 36, 237.) The capitula of *Matricaria chamomilla* yield in addition to a deep blue essential oil, which deposited a very small amount of umbelliferone methyl ether, the following compounds:—(i.) Salicylic acid, together with, apparently, an

octoic acid; (ii.) apigenin,  $C_{15}H_{10}O_5$ ; (iii.) a glucoside of apigenin, which could not be obtained in a crystalline state; (iv.) umbelliferone methyl ether,  $C_{10}H_8O_3$ , and a crystalline product (m.p.  $237^{\circ}$ – $239^{\circ}C.$ ) which possessed the characters of a mixture of umbelliferone and a dihydroxycoumarin; (v.) choline,  $C_5H_{15}O_2N$ ; (vi.) triacontane,  $C_{30}H_{62}$ ; (vii.) a phytosterol,  $C_{27}H_{46}O$ ; (viii.) a phytosterol glucoside,  $C_{33}H_{56}O_6$ ; (ix.) palmitic, stearic, cerotic, oleic, and linolic acids, together with an indefinite mixture of volatile fatty acids. A quantity of sugar which yielded *d*-phenylglucosazone (m.p.  $208^{\circ}$ ) was also present. In addition to these constituents the flower heads yielded 5.9 per cent. of fatty and resinous substances. (See also *Y.B.*, 1913, 228.)

**Mesembryanthemum expansum and M. tortuosum, Examination of.** C. Hartwich and E. Zwick y. (*Apoth. Ztg.*, 1914, 29, 925, 937, 949, 961; *Chem. Abstr.*, 1915, 9, 1092.) The sample consisting of the entire plant (roots, stems, leaves and fruit), known as channa, was shown to contain among other principles, citric and phosphoric acids, Mg, an alkaloid and a wax. The alkaloid, *mesembrine*,  $C_{16}H_{19}NO_4$ , is precipitated by the usual alkaloidal reagents, dissolves readily in  $CHCl_3$ , EtOH and acetone; sparingly in  $Et_2O$ , still more so in petroleum ether and  $C_6H_6$ , very slightly in  $H_2O$  and alkalies. The best colour test is obtained with V +  $H_2SO_4$ , the solution developing a brown-red with a greenish cast, the latter becoming distinct on warming; on standing 24 hours the colour becomes a pure bright green. The substance (occurring in the leaves to the amount of not more than 0.3 per cent. and in the roots and stems 0.86 per cent.) is phenolic in character and unsaturated. Physiologically it is in some respects not unlike cocaine, although marked differences exist. Attention is directed to a possible similarity between cocaine and mesembrine, the latter differing from the former by  $-CH_2$ . The wax of channa smells weakly aromatic, softens at  $65^{\circ}$  and melts completely at  $82^{\circ}$ . It has sp.g. 1.002, acid value 27.57–25.89, saponification value 160.11–160.78, ester value 133.71, I value 27.30–25.50. On treatment with alcoholic KOH and subsequent acetylation, 2 compounds were obtained, a hydrocarbon *mesembrene*,  $C_{28}H_{56}$  (acetate,  $C_{30}H_{50}O_2$ , m.p.  $66^{\circ}$ – $67^{\circ}C.$ ), rhombic plates m.p.  $68^{\circ}$ – $69^{\circ}C.$ , and an alcohol *mesembrol*,  $C_{31}H_{64}O$  or  $C_{30}H_{62}O$ , m.p.  $73^{\circ}$ – $74^{\circ}C.$

**Metanartheicum luteoviride, Chemical Investigation of.** G. Ueda. (*Jap. Pharm. Journ.*, 1914, 389.) This Liliaceous

plant, long known as a diuretic, is shown to contain a glucoside. This was extracted with EtOH, and purified by precipitation with basic lead acetate, and then with tannic acid. The tannic acid precipitate was dissolved in EtOH and the solution evaporated dry with ZnO. The residue was extracted with absolute EtOH and the solution evaporated to dryness. The glucoside was so obtained as a light yellow, transparent, bitter mass, which did not reduce Fehling's solution. After boiling with dilute HCl it yielded a brown, ether-soluble substance and a solution which reduced Fehling's solution.

**Ornithoglossum glaucum** Bulbs, Investigation of. (*Bull. Imp. Inst.*, 1915, 13, 59.) The dried bulbs of the Cape Slangkop contain neither alkaloids nor cyanogenetic glucosides, but yield a toxic bitter principle not yet isolated pure, but which is very poisonous. The plant, therefore, should be removed from all grazing land.

**Paullinia pinnata** Leaves, Constituents of. (*Bull. Imp. Inst.*, 1915, 13, 47.) Used under the name of "Ebonka leaves" by the natives of Sierra Leone for stomachic disorders. They prove to contain no active principle, and the small quantity of crystalline alkaloid isolated could exert no medicinal action. The various extracts of the drug were devoid of physiological action.

**Pentaclethra macrophylla** Bark, Constituents of. (*Bull. Imp. Inst.*, 1915, 13, 47.) The bark, known as "Faibeau tree" bark, is used by the natives of Sierra Leone as an anthelmintic and as a fish poison. It is found to contain no alkaloid or glucoside; but a crystalline neutral principle was isolated from it. This was physiologically inactive. Tannin to the extent of 7.1 per cent. was present. The poisonous alkaloid *paucine* was isolated in 1894 by Merck from the seeds of the tree. The bark is of no value in European medicine.

**Phytolacca abyssinica** Fruits, Examination of. R. K u e n y. (*Arch. Pharm.*, 1914, 252, 350; *Chem. Abstr.*, 1915, 9, 121.) The following substances were isolated: (1) A fat which can be separated into an oil and a solid fat and which contains a resinous material. (2) A phytosterol-like, unsaturated alcohol. (3) A hydrocarbon. (4) Two volatile acids. (5) A saponin yielding on hydrolysis an amorphous prosapogenin, dextrose, fructose and galactose. (6) A tannic acid. (7) A slime yielding galactose and pentose. (See also *Y.B.*, 1914, 210.)



**Plant Chemistry, Notes on.** P. Q. Keegan. (*Chem. News*, 1914, 110, 211.) An extract of Grass of Parnassus (*Parnassia palustris*), made with boiling water, contained a moderate amount of mucilage and no nitrate. It gave reactions of a tannoid, probably rutin, and also of a catechol tannin (2–3 per cent.). Butterwort (*Pinguicula vulgaris*) contained no nitrate and no tannin, but considerable quantities of mucilage, sucrose, and a tannoid apparently resembling quercitagenin and associated with a catechin-like substance. Golden saxifrage (*Chrysosplenium*) yielded mucilage, some gum and sucrose, a little nitrate and tannin (a true catechol without any pyrogallol derivative) but no tannoid; there was also present a substance with a quinol group (1 : 4). Leaves of mahonia or holly-leaved barberry resembled those of common ivy, in containing caffetannin and a tannoid, probably quercitagenin, and in giving certain reactions, e.g., aqueous extracts, on addition of dilute alkalis, produced a fine green coloration tending to become brown, and reddened by acids—a reaction rare in plant chemistry. The monkey flower (*Mimulus luteus*) contained nitrate, about 1 per cent. of caffetannin, a bitter principle soluble in  $\text{H}_2\text{SO}_4$  and in  $\text{C}_6\text{H}_6$  with purplish colour, an unknown substance yielding a green colour and precipitate with  $\text{HCl}$  and  $\text{HC}_2\text{H}_3\text{O}_2$ , but no rhinanthin nor tannoid. Very small quantities of caffetannin were detected in the leaves of the *Hemerocallis* and the turk's head or martagon lily. Analyses of forest leaves at different stages of growth revealed the fact that in the young leaf tannoids alone are frequently produced, and only later do tannins appear. The tannoids thus seem to be products of alkaline protoplasm, and they are associated with volatile oils, resins and waxes, whilst the tannins are associated with organic acids, especially oxalic acid.

**Podophyllum emodi, Wild and Cultivated.** (*Bull. Imp. Inst.*, 1915, 13, 37.) The Indian rhizome contains more "podophyllin" than the American drug, and all the work hitherto done on the Indian rhizome has been concerned with the wild plant.\* Two samples of *P. emodi* cultivated in India have recently been examined. These contained 12·3 and 13·2 per cent. of total resin, calculated on the dry material; the podophyllotoxin amounted to 5·2 and 3·5 per cent. A recently received sample

\* The original material reported on by J. C. Umney (*Y.B.*, 1892, 395) was derived from plants cultivated at Kirkcaldy, Scotland, by the late John Sang.—*Ed. Y.B.*

of wild Indian podophyllum gave 16.0 per cent. of resin in the rhizomes and 14.2 per cent. in the separated rootlets. The mixed resin from both gave podophyllotoxin equivalent to 2.5 per cent. on the dry material. The wild drug therefore contains more "podophyllin" but less podophyllotoxin than the cultivated specimens.

**Poisonous Woods.** H. Matthes and E. Schreiber. (*Ber. Pharm.*, 1914, **24**, 385-444; *Chem. Abstr.*, 1915, **9**, 1661.) Royal teak or Moah wood (*Illipe longifolia* or *I. latifolia*); Teak (*Tectonia grandis*); Teak (*Flindersia australis*); Lapacho wood (*Tecoma araliacea*) and Greenheart wood (*Bignonia leucoxylon*) were examined. (1) Royal teak or Moah wood contains 0.7 per cent. lapachonone,  $C_{18}H_{16}O_2$ , m.p.  $61.5^{\circ}C$ ., heretofore reported only in lapacho wood by Crosa and Manuelli. It yields the picrate,  $C_{18}H_{16}O_2 \cdot C_6H_3O_7N_3$ , m.p.  $153^{\circ}C$ . Further, yellow crystals of lapachol,  $C_{18}H_{14}O_3$ , to the amount of 0.1 per cent., also reported only in lapacho wood by Paterno, and by Stein in greenheart wood. In addition, 3 resins were isolated: an  $Et_2O$ -soluble variety (a) 0.76 per cent.;  $CHCl_3$ -soluble (b) 1.03 per cent., and  $EtOH$ -soluble (c) 3.35 per cent. The poisonous effects are due neither to lapachol nor lapachonone but rather to the resins, of which the  $EtOH$ -soluble resin is the least toxic. No glucoside, glucoresin nor alkaloid was detected in this wood. The following tests were applied in the recognition of Moah wood: (a) *Lapachol test*: On the application of about N/10 KOH, small cherry-red dots appear on the surface; (b) *Lapachonone test*: If one of the colourless crystals from the woody tissues be placed on strong  $H_2SO_4$ , an intense indigo blue colour results. (2) *Tectonia grandis* (Teak wood, Djati). While Romanis reported the presence of tectoquinone in this wood, none was found in the present instance. In addition to a little oil and a hydrocarbon of the formula  $C_{17}H_{24}$ , 3 resins were isolated by fractional treatment with solvents, characterized as follows:  $Et_2O$ -soluble (a) 0.55 per cent.;  $CHCl_3$ -soluble (b) 1.15 per cent.;  $EtOH$ -soluble (c) 2.93 per cent. All three resins produce dermatitis accompanied at intervals by intense itching. Lapachol and lapachonone were not detected. (3) *Flindersia australis* (Native teak, Moah wood not identical with that first described) contains, in addition to a resinous oil, resins separable by fractional extraction. Resin oil 6.07 per cent.;  $Et_2O$ -soluble resin 7.40 per cent.;  $CHCl_3$ -soluble resin 3.36 per cent.;  $EtOH$ -

soluble resin 2.83 per cent. An alkaloid, *flindersine*,  $C_{23}H_{26}O_7N_2$ , m.p.  $182^{\circ}$ – $183^{\circ}C$ . was obtained. It is insoluble in water, soluble in  $CHCl_3$ ,  $EtOH$ ,  $C_6H_6$ ,  $AcOH$ ,  $HCl$ ,  $H_2SO_4$ , caustic alkalies, glycerol, paraffin and fatty oils, difficultly so in petroleum benzine. It is optically inactive, unites with  $Br$  to yield the addition-product,  $C_{23}H_{26}O_7N_2Br_2$ ; with picric acid and  $PtCl_4$  to form crystalline salts, and with the usual alkaloidal reagents to yield precipitates. None of the constituents isolated from this wood were found to irritate the skin or otherwise possess toxic properties. (4) Lapacho wood contains 0.26 per cent. of lapathonone and 1.93 per cent. lapachol in addition to resins which in contact with the skin produce mild irritation and are as follows:  $Et_2O$ -soluble 0.91 per cent.;  $CHCl_3$ -soluble 2.33 per cent.;  $EtOH$ -soluble 8.36 per cent. Petroleum-benzin-soluble resin extract, amounting to 3.29 per cent., produces no irritation when brought into contact with the skin; the other 3 resins produce a marked reddening of the skin but no itching sensation, the inflamed localities healing completely after 6 days. (5) *Tecoma araliacea*. This very hard and heavy wood contains, in addition to 7.64 per cent. lapachol, resins separable by fractional extraction and capable of producing intense itching and inflammation of the skin, being characterized as follows:  $Et_2O$ -soluble resin 1.49 per cent.;  $CHCl_3$ -soluble resin 1.55 per cent.;  $EtOH$ -soluble resin 5.02 per cent. (6) Greenheart wood contained 3.69 per cent. of lapachol and resins separable by solvents. These are  $Et_2O$ -soluble resin 3.67 per cent.;  $CHCl_3$ -soluble resin 1.32 per cent.;  $EtOH$ -soluble resin 5.92 per cent. The irritant action of all these woods is due to the resins, and more particularly to the unsaturated fatty acids therein. All woods derived from the N.O. Bignoniaceae, to which *Tecoma* and *Illipe* belong, contain, besides lapachol or lapathonone or both, resins which are more or less irritant to the skin. There appears to be no ground for the assumption that the resins are derived from lapachol or lapathonone.

**Potentilla tormentilla, New Crystalline Constituent from.** A. Goris and C. Vischniac. (*Comptes rend.*, 1914, 160, 77.) The dried powdered root of *Potentilla tormentilla* was moistened with basic lead acetate and the mass extracted with boiling acetone. The concentrated acetone solution when cold deposited various  $Pb$  compounds and separated a yellow oil. These were removed, and the acetone solution was treated

with a large volume of water. The precipitate thus obtained was redissolved in EtOH 90 per cent., purified with  $\text{Pb2C}_2\text{H}_3\text{O}_2$ ; excess of the salt removed with  $\text{H}_2\text{SO}_4$  1 : 10; and the filtrate concentrated under reduced pressure. The precipitate thus obtained is recrystallized from dilute EtOH. The product is *tormentol*, a new ester-alcohol,  $\text{C}_{33}\text{H}_{50}\text{O}_{10}5\text{H}_2\text{O}$ , forming radiating groups of white acicular needles;  $[\alpha]_D + 10^\circ 78'$  in EtOH 90 per cent. calculated for the anhydrous substance. Its optical activity varies with the solvent; even when dehydrated over  $\text{H}_2\text{SO}_4$  *in vacuo*, its deviation is greater than can be accounted for by mere loss of water, and is greater still on heating. Tormentol appears to be readily polymerized. Very soluble in EtOH, MeOH and  $\text{C}_3\text{H}_6\text{O}$ ; insoluble in  $\text{Et}_2\text{O}$ ,  $\text{CHCl}_3$ ,  $\text{C}_6\text{H}_6$ , and water. When saponified it liberates an acid and an alcohol, neither of which have been crystallized. The former melts at about  $280^\circ\text{C}$ ., the latter near  $310^\circ\text{C}$ . Tormentol is readily esterified by organic acid anhydride, but the reaction mixture contains many secondary products. It affords no evidence of a Ketonic function in its molecule.

**Rhagodia hastata, Occurrence of Trimethylamine and its Origin in the Australian Salt Bush.** R. W. Challinor. (*J. Roy. Soc., New South Wales*, 47, 236; *Chem. Abstr.*, 1914, 8, 2454.) *Rhagodia hastata*, known vernacularly as "salt-bush," is indigenous in Australia and cultivated extensively as an ornamental hedge. It has long been noted for the peculiar and objectionable herring-brine odour which it gives off at certain times of the year, when crushed between the fingers. Trimethylamine was found in the distillate after distilling the salt bush with caustic alkali. It is probably derived from the decomposition of a basic substance allied to choline.

**Saffron, Scheme for Analysis of.** G. Fromme. (*Apoth. Zeit.*, 1914, 29, 737.) After enumerating the known adulterants of saffron, the author gives the following method of analysis. From 0.2 to 0.3 Gm. of the sample is weighed in a tared Erlenmeyer flask, covered with 25 c.c. of petroleum ether, and the weight noted. The flask is then attached to a long tube condenser, and the contents just boiled for 15 minutes. After cooling, the original weight is made up with more petroleum ether. After mixing and settling, exactly 20 c.c. of the liquid is removed, and passed through a tared filter into a tared dish, the filter being washed with a little of the solvent. The filtrate

is then cautiously evaporated, and dried to constant weight. The oily residue should not exceed 5 per cent. The petroleum ether remaining in the Erlenmeyer is then evaporated by immersing the flask in hot water. The residual saffron is then treated with EtOH 90 per cent., 20 to 25 c.c., and again heated under the tubular condenser for 15 minutes. The EtOH solution is then filtered through the same filter and wash this with 10 and 10 c.c. of EtOH. Wash back any saffron which may have collected in the neck of the Erlenmeyer with 20 c.c. of water. Add another 10 c.c. of water and 3 c.c. of AmOH and again heat in a boiling water-bath for 15 minutes under the reflux tube. Then filter through the tared filter, and wash it several times with hot water. Return any fragments of saffron to the flask, using 30 c.c. of water for this purpose. Add 3 c.c. of 25 per cent. HCl to the saffron and water, and heat as before for 15 minutes. Pass the liquid through the tared filter and wash all the insoluble matter on to it by means of hot water. Continue the washing first with water, then with EtOH, and finally with a little Et<sub>2</sub>O. Dry first in the air, then at 100°C. to constancy. Pure saffron will yield 16 per cent. of insoluble matter by this method, of which 4.8 per cent. (0.77 per cent. of the pure saffron) is sandy ferruginous, partially insoluble ash.

To determine the amount of added sugar, introduce 0.5 Gm. of the saffron into a small graduated tube. This should be 6 mm. inner diameter, closed at one end, and graduated at 5.1 c.c. From 1 to 1.5 c.c. of water is added, and the saffron shaken up therewith. Water is then added to the 5.1 c.c. mark; since 0.5 Gm. of saffron contains about 0.1 Gm. of water-insoluble matter. Cork, shake up, and heat for 10 minutes in the nearly boiling water-bath. Allow to settle, or centrifugate. Transfer 0.5 c.c. (=0.05 Gm. of saffron) of the clear liquid with a little yeast and a trace of tartaric acid to a saccharometer. Pure saffron should indicate from 6 to 7 per cent. of fermentable sugar. Any amount above 7 per cent. must be regarded as added sugar. (See also *Y.B.*, 1911, 232; 1912, 69; 1913, 293; 1914, 44.)

**Sarcocephalus esculentus** Roots, Constituents and Medicinal Value of. (*Bull. Imp. Inst.*, 1915, 13, 46.) Under the name of "Egbesi" this drug is considered to be a tonic and antiperiodic in Sierra Leone whence the specimens were sent. The bark of the stems or branches had previously been examined and stated to contain three alkaloids, one of which was named doundakine.

The drug was stated to be a valuable antiperiodic and was named "African cinchona." Heckel and Schagdenhauffen, however (*Y.B.*, 1886, 175), stated that they were unable to detect any alkaloidal constituent in the bark, and that whatever activity it possessed was due to a yellow resin, and that it can in no way be regarded as a substitute for cinchona. The roots examined at the Imperial Institute are also found to contain no crystalline substance. Two resinous constituents were isolated, but neither of these had any marked physiological action. The drug is not considered to be of any medicinal value.

**Scutellaria altissima, Scutellarein from.** G. Bargellini. (*Gaz. Chim. Ital.*, 1915, 45, I., 69; *J.S.C.I.*, 1915, 34, 271.) Scutellarein,  $C_{15}H_{10}O_6$ , is formed, together with glucuronic acid, by the hydrolysis, with 30-40 per cent.  $H_2SO_4$ , of scutellarin,  $C_{21}H_{18}O_{12}$ , a glucoside found in *Scutellaria altissima* and other species of *Scutellaria*, *Galeopsis*, and *Teucrium*. Natural scutellarein is identical with a tetrahydroxyflavone synthesized from 2.3.4.6-tetramethoxyacetophenone and methyl anisate by Kostanecki's method. This proves that scutellarein must be either 1.3.4.4'- or 1.2.3.4'-tetrahydroxyflavone, in agreement with the alternative formulæ suggested by Goldschmiedt and Zerner. (See also *Gen. Index*.)

**Securidaca longipedunculata, Investigation of.** (*Bull. Imp. Inst.*, 1915, 13, 50.) This Polygalaceous shrub, distributed throughout tropical Africa, yields the Buaze fibre. The leaves are stated to be used, as an infusion, for venereal diseases. The bark is employed as a stomachic. In Zambesia and Sierra Leone the leaves are used as a snake-bite remedy and the root bark instead of soap. Chemical analysis show that the roots contain about 0.1 per cent. of methyl salicylate and 4 per cent. of a saponin.

**Taxus cuspidata, Constituents of the Leaves of.** R. Ueda and K. Haseda. (*Jap. P. J.*, 1914, 388; *Chem. Abstr.*, 1915, 9, 623.) The amorphous alkaloidal substance extracted from the leaves was separated into 2 components, by the fractional crystallization of the chloroaurates. The difficultly soluble chloride crystallized from EtOH in yellow leaflets, m.p. 68°, while the more readily soluble one formed an amorphous yellow powder, m.p. 125°. The free bases could not be crystallized.

**Tephrosia vogelii from Rhodesia, Investigation of the Leaves and Seeds of.** (*Bull. Imp. Inst.*, 1915, 13, 61.) The leaves of the

Rhodesia plant were found to contain the same active constituents as were isolated by Hanriot from the Malagasy fish poison (*Y.B.*, 1907, 159). These occurred in the following percentages: tephrosin, 0.15; tephrosal, 0.06; yellow substance, 0.05. The last is crystalline and melts at 228°–229°C. The seeds were also examined. They contained twice as much tephrosin as the leaves and the same yellow crystalline substance. Another crystalline non-nitrogenous substance melting at 109°C. was also isolated from them. Probably the seeds would be even more active as a fish poison than the leaves.

**Tetracera obtusata Leaves, Constituents of.** (*Bull. Imp. Inst.*, 1915, 13, 48.) The above is believed to be the botanical source of the leaves examined, known in Sierra Leone as "Anet Leaves." Although attributed with medicinal value by the natives, no active principle except a trace of a saponin were detected. The drug has no medicinal value.

**Tetrapleura thoningii Fruits, Investigation of.** (*Bull. Imp. Inst.*, 1915, 13, 49.) Of the dark brown pods of this Leguminous plant from Nigeria, some were filled with soft sugary pulp, others contained no pulp. Neither pods nor seeds gave any cyanogenetic glucosides or alkaloids. The sugary pulp contains a saponin.

**Tomatoes, Effect of Conditions on the Acidity of.** B. M. Duggar and M. C. Merrill. (*Ann. Missouri Botan. Gard.*, 1914, 1, 237–40; *Chem. Abstr.*, 1915, 9, 1345.) With fruit of the same variety grown under the same conditions, the total acidity of green, ripening, and ripe fruit was found to be very nearly uniform, from 0.57 to 0.58 per cent. of citric acid. Fruit ripened in an incubator at 32°–33°C. for 10–22 days showed higher acidity than fruit ripened on vines or at the laboratory temperature. Normally ripened fruits of the yellow varieties were found to contain as much acid as the red. No relation was found between total acidity and pigmentation. (See also *Y.B.*, 1905, 144, and *Gen. Index.*)

**Treculia africana Fruit and Leaves, Investigation of.** (*Bull. Imp. Inst.*, 1915, 13, 64.) The leaves and fruit of the tree are reputed in Lagos to be poisonous to animals. Aqueous and EtOH extracts of both these, prepared in Lagos and forwarded to this country, are found not to be poisonous.

**Warburgia ugandensis** Bark, Investigation of. (*Bull. Imp. Inst.*, 1915, 13, 50.) The drug is used in the East African Protectorate as a purgative. It contained no alkaloids, about 3 per cent. of mannitol, and an amorphous resinoid substance to which the pungent taste is due. The drug is known locally as "Olsogonoi" or "Muziga" bark.



# MATERIA MEDICA

## NEW REMEDIES

(To June 30, 1915)

COMPILED BY THOS. STEPHENSON, F.R.S. EDIN.

[*Note*.—As far as possible, reference to products of German origin has been omitted. Where such are referred to, it is usually with the object of indicating a substitute.—COMPILER.]

**Alkagen** is a tablet consisting of magnesium hydroxide and oil of peppermint. Introduced as an antacid that does not give rise to  $\text{CO}_2$ .

**Antalgine**.—*See* **Tocanalgin**.

**Antiformin** is the trade-name of a German disinfectant, consisting of an alkaline hypochlorite to which an alkaline hydrate has been added. Used in bacteriological work on account of its solvent powers on all except acid-fast organisms. An efficient substitute may be made from the following formula:  $\text{K}_2\text{CO}_3$ , 58 Gm.; chlorinated lime, 80 Gm.; water to 1,000 Gm. Mix the chlorinated lime with 400 Gm. of the water; dissolve the  $\text{K}_2\text{CO}_3$  in 300 Gm. of boiling water and pour the hot solution into the chlorinated lime mixture. Shake the flask, stopper it, and set aside to cool. Then add sufficient water to make the contents weigh 1,000 Gm. Mix this solution with an equal quantity of a 15 per cent. solution of  $\text{NaOH}$ .

**Aurocantan**.—German name for *Gold-Cantharidin*, *q.v.*

**Calmonal** is methane-calcium bromide,  $\text{CaBr}_2 \cdot 4\text{CO}(\text{NH}_2)\text{OC}_2\text{H}_5 + \text{H}_2\text{O}$ , a crystalline powder containing 27 per cent. of bromine, easily soluble in water or alcohol. Given in doses of 1 to 2 Gm. in mild and medium cases of insomnia, particularly in the aged.

**Coagulen** is a hæmostatic obtained by the precipitation of blood, and is believed to contain the actual coagulating principle

of blood. The dry powder may be used locally for wounds, or may be given internally in cases of hæmorrhagic ulcer. The solution (6 to 12 per cent. in water) may be applied locally, or may be injected intravenously to reinforce the coagulating power of the blood. Also known as *Euclottin*. (See also **Coagulose**, *Y.B.*, 1914, p. 156.)

**Colloidal Strychnine**.—When strychnine is precipitated from solution by means of Lloyd's reagent (a variety of aluminium silicate) a colloidal compound is formed, which contains 6.15 per cent. of the alkaloid. In this compound the bitter taste of strychnine is almost completely masked, its action is considerably retarded, and its toxicity correspondingly reduced. These facts suggest kaolin or fuller's earth as a useful antidote to strychnine poisoning. (See *Y.B.*, 1914, 1, 11.)

**Colresine** is a wound varnish similar to **Mastisol**, *q.v.*

**Cuprentum** is an ointment containing 5 per cent. of soluble copper citrate, for use in trachoma and other eye affections.

**Cystazol** is a combination of hexamine and sodium benzoate which raises the acidity of the urine and favours liberation of formaldehyde.

**Diarsenol** is the name under which arsenobenzol ("606") is produced in Canada.

**Enomorphine** is allyl-morphine sulphate. The action of morphine is said to be modified by introduction of the allyl group, with the result that this compound is only slightly narcotic. Enomorphine acts as a mild stimulant to the respiration, and is said to be much less dangerous than morphine.

**Euclottin**.—See **Coagulen**.

**Fluorescein-Zinc** is obtained by the double decomposition of fluorescein-potassium and  $\text{ZnSO}_4$ . It is a reddish-yellow powder, soluble in 1,000 parts of water, forming a fluorescent liquid which gives no reaction for zinc. It is used either dry or in solution as an application in conjunctivitis.

**Formoglyceral** is an elixir of glycerophosphates of calcium, sodium, and iron, with formates and formic acid, without strychnine.

**Gold-Cantharidin** or cantharidin ethylene-diamine-aurocyanide

(German trade-name *Aurocantan*) is a combination used in the chemotherapy of tuberculosis. Dose, 0.025 to 0.1 Gm. of intravenous injection.

**Hepol** is a compound of cod-liver oil with the alkaloidal extractives of cod's liver tissue.

**Hetroform** is a urinary antiseptic composed of hexamine and benzoic acid.

**Hirudin** is the active principle of a secretion derived from the buccal glands of the leech. It acts as an anticoagulant, and its use has been recommended in blood-transfusion as being superior to sodium citrate in that it does not cause decalcification of the blood. It has also been used in eclampsia (see *Y.B.*, 1913, 244). It is sometimes spelt *Herudin*. (See also *Y.B.*, 1905, 196.)

**Hydrentum** is the name given to a yellow oxide of mercury ointment for ophthalmic use, prepared in various strengths from  $\frac{1}{2}$  to 5 per cent.

**Iodatol** is an iodine compound designed to replace the German product "Iodipin." It is made in three strengths, 10, 20, and 25 per cent. respectively.

**Iodobenzine** is an antiseptic liquid prepared by the addition of tincture of iodine to "benzine"; the excess of tincture sinks to the bottom, and a saturated solution of "iodobenzine" is formed in the top layer.

**Iodogen** is a liquid organic compound of iodine, containing the equivalent of one grain of iodine per fluid drachm.

**Iodized Carbon** is a molecular combination of iodine and animal charcoal used for the dressing of wounds.

**Iodocol** is a powder containing 1 : 30 of free iodine. It is prepared by dissolving 10 Gm. of iodine in ether, mixing the solution with 300 Gm. of sterilized kaolin, and drying. It is used as a wound dressing, and for the extemporaneous preparation of tincture of iodine. (See also **Quinolodol**.)

**Kharsivan** is the name given to a brand of British-made arsenobenzol ("606").

**Laxase** is a form of agar-agar in tablets, used for constipation.

**Lueticin** is a killed culture of *Treponema pallidum*, used as an intracutaneous test for syphilis.

**Mastisol** is a German surgical varnish used for wounds. A substitute may be made from the following formula : Mastic (finest), 40 Gm. ; benzene (pure), 60 Gm. ; castor oil, 20 drops.

**Mycetol** is the name given to a British substitute for "Lysol."

**Olaxine** is a paraffin jelly, containing 95 per cent. of paraffin, used for constipation. *Osacol* is a similar preparation containing 75 per cent. of paraffin.

**Oxidol** is the trade-name for a specially pure hydrogen peroxide.

**Oxyplnene** is a vapour produced by the passage of pinene vapour through a current of ozone. It is used in respiratory affections.

**Pavimol** is a 50 per cent. emulsion of poppy-seed oil. Given as a substitute for cod-liver oil.

**Quinacol** is a chemical combination of quinine with guaiacol, containing 50 per cent. of each. Put up in capsules of 4 minims, and recommended for phthisis, whooping-cough, etc.

**Quiniolodol** is a combination of iodine and cinchona, recommended as a substitute for iodoform in wound dressing. It can be made of any desired strength by the following process : For a 5 per cent. preparation, 5 Gm. of iodine is dissolved in 100 c.c. of ether ; 100 Gm. of powdered cinchona bark is added, and the mass triturated until all the ether has evaporated. The resulting mixture is sifted so as to form a fine powder. Quiniolodol is said to have an agreeable odour and to be powerfully antiseptic.

**Sanusin** is a suppository containing resorcin, boric acid, balsam of Peru, zinc and bismuth carbonate, designed as a substitute for the German product "Anusol."

**Seroden** is a combination of iodine with serum proteins, offered as an organic iodine compound. Capsules contain 6 grains each, equivalent to one grain of iodine.

**Silver-Arsenobenzol** is a compound of arsenobenzol ("606") with silver bromide ; recommended for use in trypanosomiasis and in syphilis.

**Silver Permanganate** has been found useful in the treatment of skin diseases, solutions varying in strength from 1 : 200 to 1 : 100,000 having been employed with benefit in facial lupus.

**Silvol** is an albuminoid compound of silver, containing about 20 per cent. of Ag. It occurs in dark lustrous scales, readily soluble in water. The solution is not precipitated by the reagents that usually affect silver salts. It is non-irritant, and may be used in solution up to 50 per cent. strength.

**Sodium-Arsenobenzol** is a golden-yellow powder, easily soluble in water, requiring no addition of alkali before use—in fact it is practically the combination that existed in the original alkalinized solution of “606” as prepared for injection. It contains 20 per cent. of As, and the dose is 0.3 to 0.45 Gm.; as much as 1.0 Gm. may, it is said, safely be given.

**Taffonal** is a preparation similar to **Mastisol**, *q.v.*

**Tocanalgin**, known also as *Antalgine* or *Morphine désintoxiquée*, was brought out recently as a non-toxic analgesic for use in obstetrics. It has since been shown to be simply morphine hydrochloride.

**Uro-hexoids** are tablets composed of hexamine and lithium benzoate. They are introduced to replace the German speciality “Cystopurin.”

## NEW APPLICATIONS OF REMEDIES

(To June 30, 1915)

COMPILED BY THOS. STEPHENSON, F.R.S.EDIN.

**Acetanilide as a Wound Application.** (*British Med. Jour.*, May 29, 1915.) Edward F. Green states that the bactericidal properties of acetanilide were first observed by him in 1891, and that since then he has used it regularly as a wound dressing, and has never noticed pus in a wound so treated. He applies the dry powder plentifully to the wound, and binds up with Gamgee tissue. The dressing is left untouched for a week, and at the end of that time the wound has been found to undergo a process of dry healing. Before application of the powder the wound is washed with hydrogen peroxide. The author suggests that if soldiers' wounds were so dressed the men could be sent home without the occurrence of inflammation or suppuration.

**Arsenobenzol, Local Application for Noma.** (*Bull. Soc. Méd. des Hôp. de Paris*, May 1, 1914.) H. Eschvach found local applications of arsenobenzol and of novarsenobenzol to be most useful in a case of buccal noma in a child of 2 years. A solution of novarsenobenzol (1 in 15) in a mixture of equal parts of glycerin and water was applied to the entire area of false membranes, the mouth was irrigated with dilute hydrogen peroxide, and hot compresses were applied to the neck. In twelve hours the membranes had loosened, and the swelling of the cheek diminished. The arsenobenzol solution was applied four times daily, and the condition gradually improved. A band of false membrane persisting on the lips, the strength of the solution was increased to 1 in 10, and then to 1 in 5, after which the last evidences of noma disappeared.

**Arsenobenzol Serum.** (*Jour. A.M.A.*, Oct. 10, 1914.) C. E. Riggs and E. H. Hammes discuss the results of 100 injections of arsenobenzol serum in 24 cases of nervous syphilis. The method employed was that of Swift and Ellis, and consists in the injection into the subdural space of 12 c.c. of serum removed from a patient who has received a dose of arsenobenzol (*New York Med. Jour.*, 1912, p. 53). On the whole they found the method satisfactory. Marked benefit followed in the tabes cases, but the results in paresis were not so satisfactory, probably on account of the advanced stage of the disease. Some improvement was noted, however, though a greater number of injections were required. Their conclusions are as follows: (1) Clinical improvement and serobiologic reduction usually go hand in hand. (2) The report of cases without associated laboratory reactions is unscientific and valueless. (3) The intraspinal use of arsenobenzol serum is a notable advance in the therapy of syphilitic affections of the central nervous system; it furnishes the one avenue of approach to the otherwise inaccessible spirochetes. (4) It is a safe procedure in the hands of trained and properly equipped men. Whether it will bear the final test of efficiency, namely, cause pleocytosis, globulin excess, and a positive reaction in the blood serum and spinal fluid permanently to disappear, is yet to be demonstrated.

**Atropine for Irritant Gas-Poisoning.** (*Lancet*, May 29, 1915.) Douglas V. Cow points out that the soothing action of atropine on the bronchioles has been made clear by Dixon and Ransom (*Jour. Physiology*, 1912, xiv., 5, 413). He has carried out a series

of experiments to ascertain its value in relieving the suffering of those exposed to German asphyxiating gases. Rabbits were subjected to the influence of chlorine and bromine for 15 minutes ; some of these were treated with oxygen, some were given injections of atropine, others were used as controls. Oxygen inhalations had little or no effect alone, but in combination with atropine injections the symptoms were relieved. The author considers that in all cases of gas poisoning atropine injections should be commenced without loss of time.

**Camphor Injections for Pneumonia.** The use of a solution of camphor in oil as an injection in cases of pneumonia has already been described (*Y.B.*, 1914, 167). The following are some recent developments of the method : O. Crouzon (*Bull. Soc. Hôp. Paris*) recommends a combination that is more readily absorbable : camphor, 1.0 Gm. ; ether, 1.0 Gm. ; olive oil, 8.0 Gm. The oil should previously have been washed with water and sterilized. The great advantage of this other combination is its ready absorbability and the absence of trouble at the site of injection. In the numerous cases in which it was used by the author, abscess formation never occurred, and only slight induration in a few cases. A. Rémond (*Bull. Acad. Méd.*) describes a camphorated serum which, he claims, is superior to camphorated oil in the treatment of pneumonia. The serum is made as follows : Camphor is digested in the cold in an isotonic serum (normal saline solution) for 24 hours. A clear liquid is obtained which is precipitated by heat, but again becomes clear on cooling, and remains clear when raised to 37°C. (98.6°F.). This serum contains about 0.2 per cent. of camphor. Injected intravenously, this serum causes rapid improvement in all symptoms arising from pneumococcic infection. The injection produces violent shivering, followed by considerable rise in temperature, after which the temperature falls to normal, with rapid improvement of clinical symptoms. The serum acts similarly in staphylococcic and streptococcic infections. Its action on the respiratory centres and on the heart is more intense than that of camphorated oil ; it produces no unpleasant effects.

**Chenopodium, Oil of, for Hookworm Disease.** One of the inconveniences occasioned by the war has been failure of the supply of thymol, a drug given in large doses for ankylostomiasis or hookworm disease. As a substitute for thymol, wormseed oil has been suggested. The value of oil of chenopodium as an

anthelmintic has already been established. It is given in doses of 8 to 16 minims, according to age, every two hours for three doses, after which a dose of castor oil is to be given. The oil acts as a paralyrant, rather than as a parasiticide, and the worms have to be expelled by purgation. Castor oil does not seem to cause absorption, as it does in the case of male fern or thymol, and is therefore the best purgative for the purpose.

**Crotalin for Epilepsy.** The employment of crotalin, the dried venom of the rattlesnake (see *Y.B.*, 1913, 240), as a remedy for epilepsy was introduced by Spangler (*New York M.J.*, Sept. 30, 1910), who tried small doses of the venom hypodermically in several cases with satisfactory results. Since then numerous investigators have reported, favourably and otherwise, on its use. Spangler (*Interst. M.J.*, Jan., 1915) reviews the literature of the subject, and describes his investigations during the five years that have elapsed since his first announcement. He himself has administered the drug in over 300 cases, and has received reports from 131 physicians who have tried it. He has also studied its composition and physiological action. Crotalin, he says, belongs to the proteins, and consists of two principal compounds, thus having a double action on the organism. The peptone element has a paralyzing effect on the nerves in large doses, while in small doses its action is that of a sedative. Its other component, a globulin, acts on the blood, in large doses completely destroying the coagulating power, in smaller doses exercising a retarding influence. Spangler finds also that the symptoms of epilepsy are closely related to certain changes in the composition of the blood, and that these changes are influenced advantageously by the use of crotalin. It has been shown that the coagulating time of the blood in epilepsy is shorter than normal, and Spangler is of opinion that crotalin has the power of lengthening this period.

The dose of crotalin should at first be not more than  $\frac{1}{400}$  grain; in children, anæmic adults, or plethoric subjects,  $\frac{1}{600}$  grain may be sufficient. A slight local reaction will result, and the second dose should not be given until this has subsided, which will be in from seven to ten days. The second dose should never be larger than the first, in order that the reaction following it may be reduced to a minimum. The best method of determining the patients' susceptibility, with a view to the regulation of further doses, is by the degree of eosinophilia produced.



The forearm is the best site for injection, and the usual precautions must be carefully observed. It is of the utmost importance that a perfectly pure and sterile preparation be used. A case has been reported by Anderson (*Journ. A.M.A.*, Mar. 21, 1914), in which the drug produced a fatal result, death evidently being due to infection from pathogenic bacteria contained in the venom. Spangler maintains, however, that with a good preparation and proper precautions, there is no such danger.

**Emetine for Cholera.** (*Indian Med. Gazette*, Jan. 1915.) Sir Leonard Rogers criticizes the results reported by Renault (see *Y.B.*, 1914, 172), who, he says, omitted the precaution of having control cases, while he began the treatment when the epidemic had reached its height, a time when a decline in the virulence would naturally take place. Moreover, Renault's cases were selected, he clearly stating that some of his assistants had given the drug in desperate cases, contrary to his express instructions. Rogers has himself tried emetine in a number of cases since March 1914, and his results force him to the conclusion that the drug has no influence for good or evil on the course of the disease when the hypertonic saline treatment is also employed.

**Emetine for Hæmoptysis.** (*Gazzetta degli Ospedali*, Dec. 20, 1914.) B. Nicola treated 21 cases of hæmoptysis in tuberculous patients by subcutaneous injections of 0.04 to 0.06 Gm. ( $\frac{2}{3}$  to 1 grain) of emetine, repeated at intervals usually of twelve hours. Analysis of the various cases shows that a good result can be counted on only when the hæmoptysis occurs in the early stages of tuberculosis, and there is not much loss of blood, or in cases with high blood-pressure and unstable vasomotor system. In all the cases of this kind the tendency to hæmorrhage was definitely arrested in from 10 to 48 hours. On the other hand, when the hæmorrhage was the result of passive venous congestion, or of an ulcerative process with abnormally low blood-pressure, emetine not only failed to arrest the hæmorrhage, but showed a tendency to induce toxic symptoms.

**Emetine for Pyorrhœa.** (*Dental Cosmos*, Aug. 1914.) M. T. Barrett and A. J. Smith state that careful examination of the gums in 46 persons suffering from pyorrhœa alveolaris in various degrees of severity convinced them that amœbæ were present in the patients' mouths, and they believe that at least

two species have been isolated in the contents of the pyorrhœa pockets. Emetine was tried in 13 cases, a half per cent. solution being injected into the pockets round the affected teeth. In several of these cases the pus disappeared completely within 24 hours after the first injection, while in every one of the cases this result was obtained after three daily injections. The tissues took on a more healthy appearance, the teeth became firmer, and the gums settled down more tightly about the roots of the teeth. No amœbæ were found after the second or third treatment, and the cure seemed to be complete, although the remedy had not been tried long enough to demonstrate positively the permanence of the cure.

C. C. Bass and F. M. Johns (*Merck's Archives*, Nov. 1914) confirm the findings of Barrett and Smith, and claim to have found active amœbæ in the lesions of pyorrhœa alveolaris. They have observed 68 cases, and have treated these with emetine administered hypodermically in doses of  $\frac{1}{2}$  to 1 grain. They inject the drug into any part of the body, but *not* into the pyorrhœa pockets, as they consider the action too local. In all cases rapid and favourable results followed.

Bacteriological examination of pyorrhœa cases has, in the experience of Leonard Rogers (*Indian Med. Gazette*, April 1915), invariably shown the presence of streptococci, and treatment with autogenous vaccines has always been satisfactory. The announcement of the discovery in America of amœbæ as the causative factor in pyorrhœa led the author to examine the pus in a case under his observation, where he found numerous active amœbæ. In other patients, however, who were doing well under vaccine treatment, no amœbæ were found. This fact, he considers, opens up an interesting field of inquiry in India, and he suggests a combination of vaccines and emetine as the most suitable treatment.

**Emetine for Sprue.** (*Jour. Amer. Med. Assoc.*, Jan. 2, 1915.) F. Schmitter has already reported (see *Y.B.*, 1914, 172) on the use of emetine hydrochloride, given hypodermically in doses of  $\frac{1}{2}$  to 1 grain, in six cases of sprue, in all of which improvement took place. Since then he has observed two more, and four more have been reported to him, making twelve cases, in all of which the patients' symptoms cleared up. He gives details of one case, and adds that no case of sprue has come to his notice which has failed to respond to the treatment.

**Emetine to Abort Typhoid Fever.** Some years ago W. L. Frazier (*Med. Record*, Mar. 20, 1915) reported on the use of ipecacuanha in salol-coated capsules to abort typhoid fever, a method which he found very satisfactory, the disease being aborted in from four to six days. The difficulty of this method, however, consisted in regulation of the dose, for at times the salol coat burst before the capsule reached the intestine, causing vomiting, while at other times the capsules passed through the tract unchanged. This difficulty has now been overcome by the hypodermic use of emetine hydrochloride. In addition to the avoidance of these drawbacks, abortion of the disease is more rapidly accomplished, taking now only from two to four days. Emetine is rapidly destructive to the typhoid bacillus. The usual adult dose is half a grain (0.032 Gm.), and injections should be started *as early as possible*, without waiting for a positive Widal reaction, and repeated every 6, 8, or 12 hours till the maximum afternoon temperature is within  $1^{\circ}$  to  $1.5^{\circ}$  of normal, after which half doses should be given till a normal temperature is reached. Emetine should not be used as an abortant after the third week of the disease, except in very small doses, as serious hæmorrhage is likely to be produced. In small doses at that stage it acts favourably but slowly.

**Ether in Surgical Therapeutics.** (*British Med. Jour.*, Feb. 6, 1915.) H. F. Waterhouse employs ether as an antiseptic in abscesses, compound fractures, carbuncles, tuberculous glands, septic wounds, sinuses, buboes, etc. In surgical work he has found it specially useful in the following conditions: (a) Infections of the peritoneal cavity, and cases in which, in the course of an abdominal operation, soiling of the peritoneum has or may have occurred. Three ounces is the maximum amount that may safely be left in the peritoneum. (b) Cases of acute arthritis proved or presumed to be secondary to a pyogenic focus existing in the body prior to the onset of the joint affection. For these two drachms is the maximum injection. (c) In the treatment of severe septic gunshot wounds. In such cases he enlarges the wound and fills it daily with ether, which is allowed to evaporate. The action of ether in these cases is complex: it acts as an antiseptic, also as a tonic on the circulation and nervous system, and it influences in some unknown way the defensive powers of the tissues.

W. W. C. Topley, in the same issue, reports on the antiseptic

action of ether on micro-organisms. In vitro, ether and its vapour have a definite bactericidal action, but this is not sufficient to sterilize the peritoneum.

**Ethyl Chloride as Local Anæsthetic.** (*Prescriber*, Sept. 1914.) W. Ford Robertson finds ethyl chloride spray to act as a perfect anæsthetic in vaccine injection. The ethyl chloride is sprayed on the part for about ten seconds, care being taken not to freeze the skin, when it will be found that the needle can be injected painlessly. He has used it in over 200 vaccine injections and has never observed any untoward effects.

**Friedmann's Tuberculosis Remedy.** The final report of the Board appointed by the Public Health Service of the United States for the investigation of Friedmann's "remedy" for tuberculosis (see *Y.B.*, 1914, 172) practically sets all doubt at rest regarding this preparation. The summary of the report contains the following statements :—

"The claim of Dr. F. F. Friedmann to have originated a specific cure for tuberculosis is not substantiated by our investigation.

"The claim of Dr. F. F. Friedmann that the inoculation of persons and animals with this organism is without harmful properties is disproved."

**Glycerin for Bromidrosis.** (*Lancet*, Dec. 5, 1914.) T. H. C. Benians finds that the application of glycerin, well spread over the soles and toes before the socks are put on, acts as an efficient preventive of bromidrosis. He used it successfully in two cases, boys of 14, in whom the trouble had persisted for several months in spite of efforts to ensure cleanliness, and in spite of the continued application of drying and antiseptic powders. He suggests its use in the army as a means of keeping the skin soft and supple.

**Hay Fever, Inoculation against.** (*New York M.J.*, Feb. 6, 1915; see also *Y.B.*, 1914, 173.) S. Oppenheimer and M. J. Gottlieb, in a preliminary report, deal fully with the literature of hay fever. The complaint is due to sensitization of an individual by the conveyance of pollen contents through the respiratory tract. Some individuals are more susceptible than others to this effect, while some are quite immune. Again, some patients are susceptible to one variety of pollen, others to other varieties. Individual susceptibility may be ascertained by the installation

into the conjunctival sac of a series of weak pollen extracts ; the one which produces congestion and swelling of the caruncle is the one to which the patient is sensitive. The authors immunize their patients by injecting gradually increased doses of pollen extract to produce tolerance to the anaphylatoxin formed in the body. The dose is increased until a local reaction appears at the site of injection ; it is continued at this amount until the reaction has ceased, and is then gradually increased as before. Eleven cases were treated in 1914, six in advance of the attack, and five during the attack. Five were cured for the season, four had the symptoms mitigated, and two received no benefit. Immunity may be determined by the complement fixation test, or from the size and intensity of the wheal produced by skin scarification, and application of pollen extract at different times before and after the treatment. The latter was the method generally adopted by the authors. As regards permanency, while immunity may not be carried over to the succeeding year, recurrences are much milder, and the reimmunizing course necessary is shorter. The best time to begin treatment is about ten weeks before the expected attack. Injections should be given weekly. Full details as to the preparation of the vaccine and technique of its application are given in the original article.

E. T. Manning (*Jour. A.M.A.*, Feb. 20, 1915) describes his treatment of the disease by injection of a solution of the pollen of ragweed (*Ambrosia* species), which is the plant usually responsible in the United States. He treated 21 cases of hay fever, and while the results were variable he reports distinct improvement in nearly all cases. He expects more favourable results next season by treating the patients before the disease begins. Treatment, he considers, must be adapted to each individual patient, and the known principles governing immunity must be thoroughly understood and applied. Moreover, certain precautions are essential : the solutions must be sterile, they must be fresh, and they must be as far as possible of uniform potency.

**Hexamine in Argyrism.** (*Jour. A.M.A.*, May 2, 1914.) A. M. Crispin describes the case of a patient who had been taking collargol internally for four years on a physician's advice, and who presented herself with a request from that physician that she be operated upon. Her complexion was of a dark bluish hue suggesting cyanosis, and certain gastro-intestinal troubles suggested that she was suffering from gall-bladder obstruction.

Careful examination, however, showed that nothing was wrong with the hepatic function, and the case was diagnosed as chronic appendicitis and argyrisms. Before operation hexamine was given in 10-grain doses, and this was found to remove the coloration of the skin most effectively. The appendix was removed, and the patient made an uninterrupted recovery.

**Iodine for Cholera.** (*Indian Med. Gazette*, Aug. 1914.) During a recent outbreak of cholera J. J. A. Brachio obtained very encouraging results from the use of iodine, which he employed in the following forms: (1) A mixture containing tincture of iodine, dilute sulphuric acid, tincture of digitalis, of each 10 minims, in one ounce of water, as a dose for adults every 4 hours; (2) a pill containing  $\frac{1}{4}$  grain iodine three or four times daily; (3) an intraperitoneal injection containing iodine  $\frac{1}{4}$  grain, potassium iodide  $\frac{1}{4}$  grain, in distilled water 20 minims. The mixture and the pills were used by subordinates in separate localities, and the results reported were very good, the former alone giving 75 per cent. of recoveries. The intraperitoneal injections were given by the author himself, and he speaks of this method as the most satisfactory of all three. Having scrubbed a small portion of the skin of the abdomen just above the cæcum, and painted the part with tincture of iodine, he injected 20 minims of the solution. In all cases adrenalin solution was given frequently, ten drops being placed on the tongue.

**Iodine Fumigation.** (*New York M.J.*, Mar. 20, 1915.) W. L. Capell describes an apparatus for the application of iodine vapour to various parts of the body. It consists of a copper cylinder, to which is attached a rubber bulb and a glass nozzle. A few crystals of iodine are placed in the cylinder, which is gently heated over a spirit lamp, and the resulting vapour is expelled from the nozzle by compression of the bulb. The advantages of iodine vapour are the shortness of time required to make the application, the evenness with which the drug is applied, and the fact that the fumes reach all parts of the cavity. Pain is reduced to a mere smarting, which lasts only a few minutes, and this smarting is less pronounced in the case of inflamed surfaces than it is on normal mucous membrane. No inflammation follows, absorption is rapid, and perfect control of the amount of iodine is obtained. The stain lasts but a few minutes.

**Iodine for Wounds: Some Suggestions.** (*Pharm. Jour.*,

*passim*.) A suggestion is made that tincture of iodine should be sprayed on the wound instead of being applied on a swab. This is not only less irritating, but has the advantage in that every crevice of the wounded surface is penetrated, while the fingers are not stained, and there is no waste of tincture. A glass atomizer must of course be used.

The irritating vapours given off from a tincture of iodine made with methylated spirit may be avoided by the preparation of a stock aqueous solution of double strength, which may be diluted with methylated spirit just before use.

**Mercurialized Serum in Syphilis.** (*Jour. A.M.A.*, Dec. 19, 1914.) C. M. Byrnes has devised a serum containing mercury albuminate, which is prepared as follows: a sufficiency of blood is withdrawn, allowed to coagulate, the serum removed and centrifugalized. To 12 c.c. of this is added 1 c.c. of a solution of mercuric chloride containing 0.0013 Gm. ( $\frac{1}{10}$  grain) in 1 c.c., and to this solution is added normal saline solution to make the volume 30 c.c. This is heated at 56°C. (132.8°F.) for half an hour, and is administered by gravity at body temperature, in the manner recommended for arsenobenzol serum. The author has found this mercurialized serum as efficacious as arsenobenzol serum, if not more so. The reactions are less severe. The great stability of this serum allows of its being prepared and kept in sealed flasks until required. (*See also Arsenobenzol Serum.*)

Lloyd Thompson (*Jour. A.M.A.*, May 1, 1915) confirms the value of mercurialized serum as described by Byrnes, and has tried it in eight cases with very gratifying results. From 40 to 50 c.c. of blood is collected and placed in a large test tube which has been boiled in salt solution. (The serum separates more rapidly and in larger quantity in a tube so prepared than in one sterilized by dry heat.) After separation, the serum is poured off and centrifugalized. To one-third of this serum is added a solution of mercuric chloride containing 0.022 Gm. ( $\frac{1}{2}$  grain) per c.c., 1 c.c. of solution being added for each 2 c.c. of serum. A heavy precipitate of mercury albuminate appears, which redissolves on addition of the remainder of the serum. The resulting mixture thus contains  $\frac{1}{2}$  grain of mercuric chloride in 7 c.c. If heated in a water-bath for half an hour at 55°C. (131°F.) and transferred to ampoules, the serum will keep indefinitely. The blood may be taken from any individual. Injections are made into the veins at the elbow with an all-glass syringe having

a sharp needle. The initial dose in all cases was 1.75 c.c. ( $\frac{1}{12}$  grain), and was increased to 7 c.c. ( $\frac{1}{3}$  grain). Symptoms of pytalism (in one case severe) appeared in several cases, but there was no occurrence of phlebitis or paraphlebitis. The syphilitic symptoms improved in all cases.

**Liquid Paraffin and Castor Oil for Chronic Dyspepsia.** (*Edinburgh Med. Jour.*, Feb. 1915.) C. M'Neil has found liquid paraffin and castor oil, given in the form of emulsions, of great value in the treatment of various and apparently distinct types of chronic dyspepsia in childhood. These types are: (a) Malnutrition, frequently associated with chronic diarrhoea, and only seldom seen with constipation; (b) enuresis with dyspeptic symptoms; (c) recurrent vomiting (cyclical or bilious vomiting); (d) recurrent attacks of fainting or sudden pallor; (e) urticaria or eczema, with dyspeptic symptoms. The emulsions of liquid paraffin are given in small, non-purgative doses of 15 to 30 minims, as may be required of the essential ingredient thrice daily. A useful adjuvant to these drugs is a powder containing one grain of calomel with sodium salicylate, 2 grains, and sodium bicarbonate, 5 grains—or with rhubarb, 1 grain, and magnesium carbonate, 3 grains—a powder being given each night. The action of the emulsions is entirely local and confined to the mucous membranes of the alimentary tract, and is probably a sedative one. The child's dietary should be carefully supervised, and the general health attended to. Notes are appended of illustrative cases in each of the groups above mentioned.

**Paraform Gauze for Wounds.** (*Bull. de l'Académie de Méd.*, Nov. 17, 1914.) Pauchet and Sourdât have found the best dressing for wounds in the field to be a gauze impregnated with paraform (solid formaldehyde or trioxymethylene  $(\text{H.COH})_3$ ). The gauze is easily prepared. A few tablets of paraform are placed under the gauze in a box, and the whole set near a stove or radiator for an hour at a temperature of about  $50^{\circ}\text{C}$ . ( $122^{\circ}\text{F}$ .). The gauze becomes automatically impregnated, and no special precautions are necessary. Paraform gauze is not only aseptic, but it sterilizes and fastens the tissues with which it comes in contact, and the wound seems to dry up without suppuration. The gauze should never be applied after tincture of iodine, as it is liable to prove irritating, but in all other cases the authors have found it the most useful dressing for wounds in war.



**Potassium Iodide for Trigeminal Neuralgia.** (*Prescriber*, July 1915.) W. D. D. Small calls attention to the variety of trigeminal neuralgia which exhibits intense pain with night aggravation. This variety almost invariably yields to large doses of potassium iodide. As a rule the disease is due directly or indirectly to syphilis, but even when this is not proved the iodide frequently acts with rapid effect. A case is cited of this nature in which 10 grains was given every four hours. Hyoscine hydrobromide  $\frac{1}{100}$  grain was used to induce sleep, and occasional doses of phenacetin were also given. The author suggests that in all severe cases the iodide should be tried.

**Quinine Hydrobromide for Whooping-Cough.** (*Jour. A.M.A.*, Sept. 19, 1914.) E. A. Hildreth speaks favourably of quinine hydrobromide as a remedy for whooping-cough. He gives 5 grains four times daily to adults, and finds it allays the spasmodic cough, relieves the sense of depression, and conduces to rest.

**Radium Applications for X-Ray Dermatitis.** (*Jour. A.M.A.*, April 24, 1915.) S. Tousey reports that he suffered for years from keratoses on his hands caused by X-ray work. Remedies such as salicylic and lactic acids had kept them under control, but the effect was only temporary. On November 11, 1914, he tried radium on one of the largest growths, 20 Mgm. in a small glass tube covered with rubber being kept almost in contact with the skin for thirty minutes. The growth was first pared down almost to bleeding point, and the field of application was limited by sheet-metal fixed by double-faced adhesive plaster. The author continues: "The result of the application was not perceptible for about ten days, then the skin became somewhat red and shiny. Twenty days more passed without any further decided change. During this whole month the pared-off keratosis had not sprung up again, as would have been the case if the radium had not been applied. On the thirty-first day, however, it looked a little doubtful, and just at the same time the reddened skin around its edges was discovered to be loosened from the underlying tissues, and in a few minutes the entire destroyed growth was removed like a scab. The surface beneath was pink and tender, but fully healed, and now, ten weeks after the radium application, the skin there is strong and presents no mark of any kind. I have been treating all the other growths seriatim. Some have been slightly blistered, but even these could scarcely

be called painful, though looking red and angry. They all come away in about a month, leaving a healthy surface beneath."

**Silver Nitrate for Wounds.** (*Presse médicale*, April 15, 1915.) J. Danysz has found from clinical observation that a solution of silver nitrate 1 : 200,000 is sufficiently antiseptic to act strongly on suppuration due to bacteria, and that, far from injuring the tissues, it markedly favours granulation and the healing of wounds. Deep, severely infected wounds are rapidly improved by a few slow irrigations, while for superficial wounds gentle spraying of the solution gives the best results. As the local condition improves the strength of the solution should be reduced to 1 : 500,000, the proliferating epidermal cells being more sensitive to the salt than those of the subjacent tissues.

**Sodium Citrate for Dysmenorrhœa.** (*Jour. A.M.A.*, Feb. 27, 1915.) B. L. Spitzig considers the causative factor in dysmenorrhœa to be viscosity of the blood, brought about by faulty hygiene, defective elimination, nitrogenous over-indulgence, and tight lacing. The stasis accompanying this viscosity causes a greater filtration of serum into the neighbouring tissues, and this induces a change in the chemical equilibrium of the endometrial cells. Nitrogenous food, which raises the viscosity of the blood, should be restricted before the menses. Sodium citrate, in doses of 20 grains three times daily during the week or two preceding the expected period, reduces the viscosity, and the author's clinical experience is that this treatment results in diminution of pain, and reduction of clots and membranes in the menstrual discharge. Nausea, dizziness, headache, and mental irritability are also lessened.

**Sodium Salicylate Rectally in Rheumatic Fever.** (*Jour. A.M.A.*, Sept. 19, 1914.) Since 1911 Louis G. Heyn has administered sodium salicylate in the form of rectal injections in cases of acute rheumatic fever. He has found this method very satisfactory, and emphasizes the ease of administration, the ready absorption of the drug, a minimum of untoward effects, ability to exhibit large doses, removal of excess of drug by subsequent enema, and prompt results, as the principal advantages. His method is as follows : A cleansing soap-suds enema is given, and after this has acted the salicylate enema is administered by means of a syringe and rectal tube inserted 6 to 8 inches. The first adult dose in men is usually from 8.0 to 10.0 Gm. (123 to 154 grains),

in women 6.0 Gm. (92 grains). The dose is incorporated in 120 to 180 c.c. (4 to 6 fluid ounces) of plain or starch water, with the addition of 1.0 to 1.5 Gm. (16 to 24 minims) of tincture of opium. The injection may be repeated in 12 hours, and if tolerated the dose may be increased from 30 to 50 per cent. daily until the limit of tolerance is reached. The largest daily dose given has been 24 Gm. (370 grains), and the only symptoms of salicylism have been tinnitus and excessive perspiration. When salicylism appears the excess of drug should be removed by means of an enema. Four cases are cited in detail, and the method is offered as a very safe and reliable solution to the therapy of this affection.

**Trichloroacetic Acid for Lupus Vulgaris.** (*Jour. A.M.A.*, Oct. 17, 1914.) M. L. Heidingsfeld prepares a saturated solution of trichloroacetic acid by adding 10 drops of water to an ounce of pure crystals. A tiny pledget of cotton is carefully wrapped around the end of a small rounded toothpick, by means of which the remedy is applied, as far as practicable, to each congested nodule. The remedy apparently exerts a selective action, attacking the nodules with greater promptness and sparing somewhat the intervening more normal or cicatrized areas. This selective action is also noted if the remedy is applied more or less diffusely to a generally congested surface, although better results are usually obtained if it is carefully applied to the individual nodules for a few seconds at a time. The application produces a momentary sense of discomfort and stinging, which can be promptly allayed by the application of a lotion containing zinc sulphide and lime water. The trichloroacetic acid should be applied to areas not much larger than a shilling, and repeated at seven- to fourteen-day intervals. Under the treatment the nodules become the seat of superficial crusts which exfoliate within from five to ten days. The general congestion diminishes, and the nodules lose in intensity of redness, and rapidly diminish in size, until they gradually become no larger than pin heads, and ultimately disappear. Mucous membrane lesions on the lips and within the nose have yielded with equal promptness and success. Details are given of a number of cases of lupus vulgaris successfully treated by this method.

## PHARMACOGNOSY

**Amomum Korarima the Greater or Korarima cardamom and its Essential Oil.** E. M. Holmes. (*Perfum. Record*, 1914, 5, 302.) A consignment of this uncommon cardamom appeared in November, 1913, at the London drug sales, where it was described as "wild cardamoms." The fruits yielded 1·2 per cent. of oil having the sp.g. 0·9038;  $\alpha_D - 3^\circ$ ; acid value, 3·6; ester value, 22·1; soluble 2:1 in alcohol 90 per cent. The odour of the oil recalls that of nutmegs rather than of cardamoms. The fruit was termed "nutmeg cardamom" by Dymock. An interesting résumé of the literature of this drug is given, which shows that it was known in Europe as early as the sixteenth century as *Cardamomum majus*, and that name was subsequently usurped by "grains of paradise." An illustration of the fruit and seeds is given.

**Amoora Rohituka Seed and its Fixed Oil.** — Weitz and — Lecoq. (*Bull. Sci. pharm.*, 1915, 75.) *Amoora rohituka* is a tree indigenous to the Malay Archipelago and Indo-China. The bark is employed by the natives as a remedy for disorders of the spleen and the expressed oil of the seeds as a liniment for rheumatism and as an illuminant and for soap making. The residual seed cake is bitter and nauseous, so it cannot be used as fodder. A full description of the fruit and seed is given. The yield of oil is about 42 per cent. It is a viscous yellowish-brown, bitter oil, with an odour resembling that of linseed oil. Sp.g. 0·929; acid value, 17·3 to 24·7; saponification value, 131·8 to 134·8.

**Belladonna Cultivated in India.** (*Bull. Imp. Inst.*, 1914, 12, 317.) *Atropa belladonna* has been grown since 1909 from imported seed at the Kutchery Garden, Naini Tal, and the yield per acre and alkaloidal content of the roots determined. The one-year-old plants yielded 3,570 lb. of roots per acre, containing 0·4 per cent. of alkaloid; the two-year-old plants yielded 3,545 lb. of roots per acre, containing 0·45 per cent. of alkaloid; the three-year-old plants gave 2,900 lb. per acre of roots, with an alkaloidal content of 0·44 per cent. The plants were found to be thoroughly established in the third year. As belladonna root imported from Europe contains from 0·2 to 0·6 per cent. of alkaloids, the samples examined are considered suitable for use in Indian medical store departments. The plant is easily grown, and is, so far, immune from insect attacks. It is believed that

in better soil, such as is obtainable in the Ramgarh neighbourhood, heavier yields of root, richer in alkaloid, should be obtained. Further experiments are being made to determine whether the drug can be grown at a profit. For this purpose additional areas have been sown with seed from acclimatized plants.

**Belladonna and Hyoscyamus in the U.S.A.** O. F. Farwell. (*Amer. J. Pharm.*, 1915, 87, 98.) Henbane is a much hardier plant in the U.S. and is established in the Straits of Mackinac in Michigan. Belladonna is hardy as far north as New York, and was established at Detroit until extinguished. Belladonna seeds sown in a garden did not germinate, but the self-sown seed germinated freely. Seeds of *Hyoscyamus bohemicus* and of *H. niger* germinate freely. Hyoscyamus plants in America are subject to destructive attacks by the potato bug, *Doryphora decemlineata* (known over here as the "Colorado beetle"), and the 3-lined potato bug, *Sema trilineata*, to such an extent that their cultivation would probably not pay. Until last year, belladonna was not attacked by insect pests.

**Belladonna Seeds, Germination of.** A. F. Sievers. (*Amer. J. Pharm.*, 1914, 86, 483.) The author records a series of experiments with belladonna seeds undertaken in the Investigation Office of Drug Plants and Poisonous Plants of the U.S. Department of Agriculture. Although the results obtained refer solely to experiments under the climatic conditions of the U.S.A. they are of interest to pharmacists in this country. Field sowing is shown to be unsuitable for the propagation of belladonna in North America. Belladonna seed germinates slowly and irregularly, and, as a rule, not more than 50 per cent. germinates at all. When once planted out, insects and weeds are the chief causes of loss. The subjection of belladonna seed to freezing temperatures accelerates their germination. Hence it is of benefit to sow the seed in the fall in order to ensure a rapid and early germination in spring. There appears to be no relationship between the size of the seed and its germinating power. A relationship does exist between the size of the seed and the vigour and strength of the plant. The heavy seeds are by far the best. The percentage of germination of the light seeds is very small. Separation of these inert seeds can be readily effected by immersing the seed in water and discarding those which do not sink. The proportion of light and heavy seed from each individual plant varies greatly. This may be due partly

to carelessness in picking the berries, as unripe berries contain light and worthless seeds. The question of drying is also of importance. The berries must be thinly scattered and dried in a well-ventilated room, in order to reduce moulding to a minimum. It is probable that a certain percentage of the seed in a fully developed berry is inert, which would account to some extent for the relatively low percentage of germination of the average belladonna seed. Colour appears to be no criterion of the value of the seed as regards germinative power. The brown seeds have a better appearance, but apparently the grey ones have equal vitality. While other investigators have found that treatment with concentrated  $\text{H}_2\text{SO}_4$  from 1 to 10 minutes is of benefit, experiments with various strengths of acid for periods ranging from 1 to 60 minutes showed that, as a whole, the treatment is not of any great value. The germination was accelerated in some instances, but no material increase in actual germination was noted. Treating seeds with  $\text{H}_2\text{O}_2$  was found to be of very material benefit. Eighteen and 24 hours gave better results than longer treatment. A 60 per cent. solution of the commercial  $\text{H}_2\text{O}_2$  gave the best results. The concentrated solution was the least beneficial. Scratching the seed coats by shaking in a bottle with powdered glass and by rubbing between sheets of emery paper, while of some benefit, was not nearly as beneficial as the  $\text{H}_2\text{O}_2$  treatment.

**Castor Oil Seeds, Detection of, in Oilcake.** G. D. Lander and J. J. Geake. (*Analyst*, 1914, 39, 292.) Active ricin is detected by incubating 2 Gm. of the material with 40 c.c. of 0.9 per cent. physiological salt solution for 1 hour at  $37^\circ\text{C}$ ., allowing the mixture to stand overnight, and then centrifuging it for an hour. One c.c. of the clear liquid is made to form a layer over about 0.1 c.c. of antiricin serum; in the presence of ricin, a zone, developing after 12 hours into a precipitate is obtained. As a confirmatory test well-washed blood corpuscles suspended in physiological salt solution are treated with the extract. Agglutination is rapidly shown with the mixture consisting of 10 per cent. castor seed and 90 per cent. linseed, but is less pronounced with smaller proportions of castor seed. Proof that ricin is the agglutinating agent is obtained by mixing equal quantities of normal serum and antiricin serum with equal quantities of the extract from the seeds, and adding to each the same amount of the blood-corpuscle suspension. Inhibition of agglutination in the antiricin mixture indicates the presence of ricin.

**Chaulmoogra Seed, True and False.** H. Pabisch. (*Pharm. Post*, 46, 889.) True chaulmoogra seeds from *Taraktogenos kurzii* contain 38 per cent. of oil, m.p. 22°–23°C., sp.g. 0.951; acid value, 23.9; saponification value, 213; I value, 103.2. It is often adulterated with false chaulmoogra oil, from *Gynocardia odorata*, *Hydnocarpus wightiana*, *H. anthelmintica*, and *H. venenata*. The seeds of the first species contain 50 per cent. oil (oleum gynocardiæ), of salve-like consistence; m.p. 22°–23°C.; sp.g. 0.952; acid value, 4.9; saponification value, 197; I value, 162.8. *H. anthelmintica* yields by pressing 17 per cent., by extraction 20 per cent. of almost colourless fatty oil, called Lukra oil or Krebao butter; m.p. 24°–25°C.; sp.g. 0.953; acid value, 75; saponification value, 212; I value, 86.4. *H. wightiana* yields 42 per cent. of light yellow oil by extraction, of butter-like consistence; m.p. 22°–23°C.; sp.g. 0.958; acid value, 3.8; saponification value, 207; I value, 101.3. The seeds of *H. venenata* are used as a fish poison. They yield an oil m.p. 22°–25°C.; sp.g. 0.955; acid value, 15.6; saponification value, 202.7; I value, 97.6. (See also *Y.B.*, 1914, 77.)

**Cinchona Barks and Adulterants, Microscopy of.** C. W. Ballard. (*J. Amer. Pharm. Assoc.*, 1915, 4, 98.) Illustrations are given of the distinctive characters of the histological elements of *Cinchona caliyasa*, *C. succirubra*, *C. ledgeriana*, *C. officinalis*, *Remijia pedunculata*, and of Maracaibo bark. The last named is used as an adulterant of cinchona.

**Clerodendron heterophyllum and some other Antisyphilitic Verbenaceæ.** Em. Perrot and G. Hubert. (*Bull. Sci. Pharmacol.*, 1914, 21, 449–52.) No alkaloid, nor glucoside capable of being split by emulsin was present in this plant, which is a native of Reunion and Mauritius, where it has a reputation as a remedy for syphilis. A full botanical and histological description of the drug is given. A small amount of an essential oil with an unpleasant odour was isolated by distillation. Other species used as anti-syphilitics are the Indian *C. phlomoides* and *C. infortunatum*, also *Vitex cymosa* and the Brazilian *Vitex montevidensis*.

**Cloves, Zanzibar, Cultural Notes on.** F. C. McClellan. (*Bull. Imp. Inst.*, 1914, 12, 415.) An interesting first-hand account of the Zanzibar clove industry.

**Drugs, Unofficial, Ash Content of.** E. L. Newcomb. (*Amer. J. Pharm.*, 1915, 87, 113.) The following percentages of

ash were given by the drugs named : *Aralia racemosa*—7·88 and 7·6. *Asarum*—9·23 and 9·22. *Baptisia*—2·298 and 2·288. *Bryonia*—A. Clean commercial sample, 6·49 and 6·17 ; B. Commercial sample powdered Bryonia, 12·51 and 12·63. (This sample, upon microscopic examination, showed numerous stone cells and other tissue not found in true Bryonia.) *Castanea*—3·927 and 3·958. *Coptis*—5·07 and 5·06 ; 4·28 and 4·91. *Convallaria flowers*—9·68 and 9·39. *Damiana*—7·40 and 7·21. *Dioscorea*—3·00 and 3·078. *Iris versicolor*—A. Commercial sample powdered in laboratory, 3·03 and 3·4 ; B. Sample consisting of the roots alone, from plants grown in the Medicinal Plant Garden, 6·00 and 5·80 ; C. Sample consisting of the rhizomes alone from plants grown in Medicinal Plant Garden, 4·77 and 4·28. *Juglans*—7·702 and 7·396. *Menyanthes*—9·56 and 9·46. *Myrica*—3·04 and 2·86. *Euphorbia*—5·76 and 5·73. *Turkey Corn Root*—4·37 and 4·528. *Chionanthus*—3·556 and 3·57 ; 3·80 and 3·50. *Fraxinus, White Ash Bark*—3·82 and 3·59. *Centaury Herb, European*—4·42 and 4·51. *Cocillana*—7·46 and 7·32. *Para Coto*—1·121 and 1·106. *Pulsatilla*—5·99 and 5·59. *Quebracho*—5·902 and 6·77. *Rumex*—A. Commercial sample powdered Rumex, 22·19 and 22·15 ; B. Commercial sample whole Rumex, select, very clean ; powdered in laboratory, 4·73 and 4·4 ; C. Commercial sample whole Rumex, ordinary quality ; powdered in laboratory, 7·7 and 7·9 ; D. Sample prepared from a plant of *Rumex crispus*, grown, 1914, in the Medicinal Plant Garden ; powdered in laboratory, 3·03 and 2·89 ; E. Sample prepared from a plant of *Rumex obtusifolius*, grown, 1914, in Medicinal Plant Garden ; powdered in laboratory, 3·33 and 3·003.

**Gum Acacia of the Anglo-Egyptian Sudan.** — All and. (*Bull. Sci. pharm.*, 1914, 21, 477.) An article, illustrated with numerous photographs, describing the collection of gum from *Acacia vereke*, and its local commerce.

**Hindoo Medicines.** E. M. Holmes. (*Pharm. J.*, 1914 [4], 39, 617.) Descriptions are given of the following Indian drugs : *Holarrhena antidysenterica*, Conessi or Tellicherri Bark ; *Plantago ovata* yielding ispagula or spogol seeds ; *Aegle marmelos*, Bael fruit ; and *Terminalia chebula*.

**Hyoscyamus and Belladonna, Morphological Investigations on.** E. L. Newcomb. (*Amer. J. Pharm.*, 1915, 87, 1.) Characteristic branching hairs on the leaves of *Hyoscyamus niger*,



*H. albus* and *Atropa belladonna* are described and drawn. Powdered hyoscyamus prepared from the dried flowering tops<sup>1</sup> when shaken with hot water, gives a faint bluish colour from the anthocyan of the flowers. When filtered, the filtrate gives a pink colour reaction with HCl. The intensity of the colour depends on the care taken in drying the drug, and the amount of basal leaves, or foreign matter. If powdered hyoscyamus is prepared from dried flowering tops the number of characteristic pollen grains is fairly constant, ranging from 766 to 1045 in 5 Mgm. of powder. If the powder is prepared from dried basal leaves, only a few pollen grains are visible. Investigation of the distribution of alkaloids in *Hyoscyamus* shows that the roots of biennial plants contain most, 0.17 per cent. ; the first year's leaves 0.069 ; second year's leaves and tops 0.068, and annual leaves and tops 0.070 per cent. A full bibliographic reference to published work on the subject is given.

**Ilex dumosa an Adulterant of Maté.** A. Lendner. (*Mitt. Lebensm. Hyg.*, 4, 42 ; *Chem. Abstr.*, 1914, 8, 2446.) The botanical examination of a commercial sample of maté (leaves of *Ilex paraguayensis*) showed that it was entirely composed of leaves of *Ilex dumosa*. It did not yield any caffeine.

**Medicinal Plant Gardens.** W. W. Stockberger. (*Amer. J. Pharm.*, 1914, 86, 506.) Under the necessary favourable conditions a fair return may be expected from several drug crops. Such crops, however, require proper horticultural treatment, and the popular and prevalent opinion that they can be grown like ordinary cultivated crops is erroneous. The Bureau of Plant Industry of the U.S. Department of Agriculture offers advice on growing drugs, and material for experimental cultivation, as well as for botanical gardens for schools of pharmacy.

**Medicinal Plants, Cultivation of.** F. A. Miller. (*J. Amer. Pharm. Assoc.*, 1915, 4, 583.) After reviewing the history of drug cultivation in the U.S.A. the author shows that the successful production of drugs, and especially the growing of European drugs in North America, is not simply a matter of sowing the seeds in the open, with subsequent field cultivation of many different species in the same soil, or under similar conditions and environment. Aconite and colchicum seeds for instance are very difficult to germinate. Convallaria grows freely in the U.S. and would be easy to cultivate. Belladonna cultivation is now

receiving a good deal of attention and appears to require certain special treatment in the American climate. In the author's experience, forced propagation and subsequent planting out is necessary. When growing and fully established, the plants are much subject to insect attacks [as they are here]. *Digitalis* grows well in Oregon and Washington and the product in those states is equal therapeutically to the European drug. *Gentian* has not yet been successfully germinated in America. Good *hyoscyamus* seed is difficult to obtain. The author has raised an annual strain of the plant which produces a quickly germinating seed. In the United States, the potato beetle is a dangerous enemy to henbane. *Stramonium* grows well and there should be no difficulty in supplying all home needs from American sources. The great aim of drug growers should be careful selection, controlled when possible by chemical analysis, so as to raise a strain of plants yielding the maximum amount of active material from a given area under cultivation. (See also *Y.B.*, 1913, 284, 285 ; 1914, 200.)

**Medicinal Plants, Cultivation of.** E. M. Holmes. (*Pharm. J.*, 1915 [4], 40, 4.) The author points out the fallacy of statements as to the profits to be derived from the growing of medicinal plants, as a commercial speculation, based on prices ruling at the present time. When the war is concluded, these prices will not be maintained. The main chance of success on normal commercial conditions is so far to improve the quality of these drugs that British grown articles should command a preference over foreign products, on their merits. The official medicinal plants, those yielding essential oils, and herbs sold by herbalists, are dealt with in the article.

**Pepper, Ground, Detection of Powdered Olive Stones in.** M. Rigotard. (*Annales des Falsifications*, 1914, 7, 132.) When olive stones are used as an adulterant of pepper, they are usually very finely ground. Consequently by grading a suspected sample the olive stone adulterant will be concentrated in the finest powder. If this be treated with paraphenylenediamine the particles of olive stones turn bright red, so that they may be picked out easily under a low micro-power or even with a lens.

**Pepper, Some New Features in the Structure of.** T. E. Wallis. (*Analyst*, 1915, 40, 190.) A full description of some histological elements found normally in ground white pepper, but differing

from those figured in works of reference. These structures are derived from pigment layers at the base and apex of the fruit. These may occasionally be sufficiently numerous to lead to the erroneous inference of foreign admixture in a genuine sample of the powdered condiment. These peculiar cells and the other structures of pepper are fully illustrated.

**Rhamnus Barks, Medicinal, Pharmacognosy of.** E. N. Gathercoal. (*J. Amer. Pharm. Assoc.*, 1915, **4**, 63, 193.) An illustrated article, dealing with the botanical characters of the shrubs and the macro- and microscopical structure of the bark of various species of *Rhamnus*. These include *Rhamnus catharticus*, *R. tinctorius*, *R. croceus*; *R. purshiana*, *R. californica*, *R. caroliniana* and *R. wightii*. The literature and history of the drugs; their pharmacopœial employment; their adulterations; published papers on the histology of the barks, and the chemical investigations are arranged in chronological order. Numerous illustrations of the microscopical characters of the various barks are given.

**Rhamnus purshiana, History, Growth, Method of Collection and Bibliography.** C. W. Johnson and Edith Hindman. (*Amer. J. Pharm.*, 1914, **86**, 387.) A very complete treatise on the subjects, including a bibliographical reference to over 140 publications concerning the bark, its history and preparations.

**Sanguinaria, Time to Collect the Rhizome.** A. O. Farwell. (*Amer. J. Pharm.*, 1915, **87**, 97.) Alkaloidal determinations made during the whole year indicate that the rhizomes are richest in alkaloids at the flowering season in May and should be collected then. (See also *Y.B.*, 1914, 203.)

**Spurious and Admixed American Drugs.** J. U. Lloyd. (*Drugg. Circ.*, 1915, **59**, 87.) A large number of American crude drugs, chiefly those used in domestic medicine, at the present time are largely mixed with spurious substitutes. This frequently occurs from careless gathering and the growing scarcity of some of the genuine drugs, rather than from wilful systematic adulteration. The following are among the chief popular samples found to be thus affected. *Chionanthus*.—Formerly this drug, the inner bark of the root, was remarkably free from substitution. Of late years, however, collectors have confused *chionanthus* with the bark of a tree or shrub, at present of undetermined species, closely

resembling the genuine article. Large parcels containing only a small proportion of genuine bark have appeared on the market. *Viburnum opulus*.—Formerly there was no difficulty in obtaining cramp bark, genuine "cramp bark," but it was gradually substituted with the bark of *Acer spicatum*. *Viburnum prunifolium*.—Black haw bark has also been freely substituted with the tasteless bark of various species of *Crataegus*. *Corydalis*.—Although only *Corydalis canadensis* is recognized by the eclectics, *C. cucullaria* is also gathered and marketed. Both species are known by the trivial name of "turkey corn" or "turkey pea." *Epilobium*.—Both *Epilobium angustifolium* and *E. palustre* are dried for "willow herb." In one instance, a large consignment sent as "willow herb" was composed of the leaves and of *Salix nigra*. The growing scarcity of *Hydrastis canadensis* has led to many admixtures with golden seal root. *Serpentaria*, *cypripedium*, *senega*, *collinsonia*, *jeffersonia* or twin-leaf, and *caulophyllum* have been found thus mixed. In some cases the admixture of blue cohosh was evidently intentional, for the pieces were cut into fragments, or the fibres only used for admixture. *Stylophorum diphyllum*, the American poppy, is sometimes gathered in mistake for *hydrastis*. It flowers at the same time and has a yellow root. White snakeroot, which should be derived from *Eupatorium aromaticum* is more often represented by the commoner *E. ageratoides*. Notwithstanding the marked difference between *helionas*, or unicorn root (*Chamaelirium*), and *Alettris farinosa* or stargrass root, the former is frequently offered indifferently one for the other because the name unicorn root is applied to both. A similar uncertainty occurs with *Apocynum*; in some districts *A. androsaemifolium* is supplied, on others *A. cannabinum*. In this case there appears to be but little difference in the therapeutic action.

## PHARMACOLOGY AND THERAPEUTICS

**Anæsthetic Liquid for First Aid.** *Schleich. (B.M.J., 1914, 2, 639.)* Ethyl chloride, 2; chloroform, 4; ether, 12; all by weight. This mixture is recommended to render unconscious the seriously wounded on the field. It is even suggested for auto-administration by the wounded man. For this purpose, each soldier going into action should be provided with a small bottle of the mixture, and a pad of cotton wool. When wounded he is to moisten the wool with the mixture and place it over the

nose and mouth, inhaling deeply. Unconsciousness quickly supervenes, followed by prolonged sleep.

**Anisol as a Parasiticide.** H. L a b b é. (*L'Union pharm.*, 1915, 56, 257.) Anisol, phenylmethyl ether,  $C_6H_5.OCH_3$ , is the most effective of all remedies tried by the author for destroying pediculi. A 1 : 20 or 1 : 40 solution in weak alcohol, sprayed on the scalp and other parts of the body infested with lice, kills the parasites and their ova at once. Undergarments and uniforms may be similarly treated with equal efficacy. The application of anisol is quite harmless to the skin. There is no danger of ignition from fire. It does not damage the fibres of textile fabrics, nor affect their colours.

**Arsenic Antidote.** O. R a u b e n h e i m e r. (*Amer. J. Pharm.*, 1915, 87, 59.) The following is claimed to be an improvement on previous formulæ:—Solution of ferric sulphate (sp.g. 1.432), 40 c.c.; magnesia magma (milk of magnesia), 300 c.c.; water, a sufficient quantity. Mix 40 c.c. of solution of ferric sulphate with 260 c.c. of water in one bottle, and mix 300 c.c. of magnesia magma, or milk of magnesia, with 300 c.c. of water in another bottle having the capacity of 1,000 c.c. When the antidote is required, add the iron solution gradually to the magnesia mixture, shake well, and the preparation is ready for instant use.

**Arsenic, Influence of, on the Growth of the Bones.** A. V a n d e n E e c k h o u t. (*Bull. Soc. Chim. Belg.*, 28, 168; *Chem. Abstr.*, 1915, 9, 225.) As promotes the formation of fat and the development of the bones in normal animals which are well nourished.

**Ascarides, Toxicity of.** A. B r i n d a. (*J. Amer. Med. Assoc.*; *Chem. Abstr.*, 1915, 9, 1509.) Intestinal round worms contain an extremely active toxalbumin. The juice expressed from them when injected causes convulsions and haemolysis. Toxic action is evident on the circulations, pulse, blood-pressure and respiration. Post-mortem examination also reveals pathological signs.

**Benzoic Acid and of its Sodium Salt, Action of, upon the Animal Organism.** E. R o s t, F. P r a n z and A. W e i t z e l. (*Arb. kais. Gesundh.*, 45, 425-90; *Chem. Abstr.* 1914, 8, 2425.) Benzoic acid and its Na salt cause vomiting in dogs on single administration. Daily administration produced a typical

poisoning, with symptoms resembling human epilepsy. On continued administration, death followed through central paralysis. No difference in action between the acid and its salt was observed. The minimal toxic dose of sodium benzoate for dogs is 1 Gm. per kilo body-weight. Glycocoll is an active antidote against benzoic acid, causing it to be excreted in the urine as hippuric acid.

**Bismuth Subnitrate, Nitrite Poisoning from.** J. Z a d e k. (*Z. exp. Path. Ther.*, 1914, 15, 498; *Chem. Abstr.*, 1915, 9, 658.) Positive reactions for nitrites were obtained in stools from a series of cases given bismuth subnitrate. Nitrite absorption and poisoning is therefore possible from the decomposition of the subnitrate in the alimentary tract.

**Bitter Tonics do not Increase the Gastric Secretion.** A. J. C a r l s o n. (*J. Amer. Med. Assoc.*, 1915, 64, 15.) Direct experiments with a case of human gastric fistula indicate that the administration of such bitter tonics as gentian, quassia, elixir of iron, quinine and strychnine, do not stimulate the flow of gastric secretion. These observations were confirmed by concurrent experiments on dogs.

**Caffeine, Action of, on the Kidneys.** G. V i n c i. (*Arch. farm. sper.*, 1914, 17; *Chem. Abstr.*, 1915, 9, 224.) The prolonged administration of caffeine exerts a harmful effect upon the urinary passages, characterized by the presence in the urine of various pathological elements, albumin, white corpuscles, more rarely red corpuscles, epithelial cells, cylinders, and corresponding modifications in the renal parenchyma. The epithelium of the dog is more sensitive to caffeine than that of the rabbit. Phenomena of epithelial degeneration precede the occurrence of pathological elements in the urine. Probably the action of caffeine is that of an epithelial diuretic.

**Calomel and Vichy Water, Therapeutic Incompatibility of.** S p i n d l e r. (*Presse méd.*; *Chem. Abstr.*, 1915, 9, 1509.) Symptoms of acute poisoning were observed in an infant after ingestion of HgCl followed by drinking of Vichy water; intoxication was probably due to a reaction between HgCl and the NaHCO<sub>3</sub> of the water.

**Cannabis sativa, Therapeutic Value of American-grown.** H. C. H a m i l t o n. (*J. Amer. Pharm. Assoc.*, 1915, 4, 448-51.)

The variable results in pharmacological tests, obtained with active material, are due to the fact that not all test animals respond uniformly to the same specimen of drug. Many of them are not sufficiently susceptible, and even susceptible ones are not uniformly so. The activity of the drug appears to reside in the leaves and the flowering tops of the pistillate plants. With the exercise of caution in selecting the drug and insistence on certain qualities essential to a drug of good quality, American growers can produce material of value practically equal to that imported from East India or elsewhere.

**Chelidonine, Action of, on the Smooth Muscles of Warm- and Cold-blooded Animals.** P. J. Hanzlik. (*Zentr. Physiol.*, 1914, 28, 551-2; *Chem. Abstr.*, 1915, 9, 104.) Chelidonine accelerates the spontaneous rhythmic movements of the œsophagus, fundus and pyloric end of the stomach of the frog, small intestine of the cat and rabbit, and the uterus of the pregnant guinea pig. It increases the activity of pilocarpine, pituitrin, histamine and  $\text{BaCl}_2$  on excised surviving organs. Perfusion with chelidonine extends the peripheral vaso-constriction in the frog caused by epinephrine more rapidly than Ringer solution. Chelidonine alone has no effect on the bore of the vessels. It counteracts the action of histamine on the bronchial musculature when perfused. Intravenous injection of chelidonine causes a diminution of the intestinal peristalsis in a rabbit, proper doses counteracting the effect of pilocarpine.

**Chrysarobin and other Medicaments used in Psoriasis, Germicidal Activity of.** J. F. Schamberg. (*J. Cutaneous Dis.*, 33, No. 1; *Chem. Abstr.*, 1915, 9, 936.) Chrysarobin and sodium chrysophanate fail to exert any germicidal effect on the *Staphylococcus pyogenes albus* either *in vitro* or *in vivo*. Pyrogallie acid fails to inhibit *Staphylococcus albus* except when the concentration is greater than 10 per cent. Arsenic (Fowler's solution) in 1 per cent. solution and sodium arsenate in 20 per cent. solution show no germicidal effect *in vitro* toward *S. pyogenes albus* with 15 minutes exposure. Fowler's solution representing 1 per cent.  $\text{As}_2\text{O}_3$  in amounts of 0.2 to 0.4 c.c. in 5 c.c. of a sterile neutral bouillon will inhibit 0.1 c.c. of a 24-hour culture of *S. aureus* after 48 hours duration. Smaller amounts fail to inhibit. The intravenous administration of 6 times the average dosage per kilo of body-weight of an  $\text{As}_2\text{O}_3$  solution does not prevent

staphylococci from producing abscesses in the kidneys. HgCl in amounts of 0.0005 to 0.001 Gm. is absolutely germicidal to the cocci contained in 0.1 c.c. of a 24-hour broth-culture of *S. aureus*. HgCl in 1 per cent. suspension will destroy cocci on both normal skin and psoriatic patches. Chrysarobin under identical conditions fails to exert any germicidal effect.

**Cicutoxin, the Poisonous Principle of *Cicuta vagans*.** C. A. Jacobson. (*J. Amer. Chem. Soc.*, 1915, **37**, 916.) The "water hemlock" of the Eastern United States is *Cicuta maculata* and *C. bulbifera* and of the Western States *C. occidentalis*. *C. maculata* appears to be identical with the European *C. virosa*. The material examined by the author was *C. vagans*, which is said to be identical with *C. occidentalis*. The toxic principle has been isolated as an amorphous resin-like substance, cicutoxin,  $C_{19}H_{26}O_3$ . A tentative structural formula for the cicutoxin molecule has been proposed. It decomposes and polymerizes readily, especially at temperatures above 50°C. It is extracted from the tubers by means of  $Et_2O$  and enters violently into combination with free bromine. It forms combinations with Pb, Ba, HCl,  $NH_3$ , and yields the double acetyl derivative.

Cicutoxin is a spasmotoxin, producing symptoms that may be separated into a prodromal, a paroxysmal and a paralytic stage. Death ordinarily results in from 30 minutes to 8 hours. The lethal dose of cicutoxin for the average rabbit is 175 Mgm., and 50 Mgm. per kilo body-weight for cats, when administered per mouth. Cicutoxin attacks a nerve centre in the calamus scriptorius and kills by asphyxiation and exhaustion. It is not a constitutional poison and the lethal dose cannot properly be given in terms of milligrams per kilo body-weight. No antidote is known for this poison, and the most reliable treatment at present consists in producing vomiting and allaying the convulsions by means of a narcotic. Cicutoxin may be detected in the  $Et_2O$  extract of the contents of the stomach by the following reaction. A 1 : 20 solution of cicutoxin when treated with a 1 : 50 aqueous solution of  $Ba(OH)_2$  added in small quantities at a time until a voluminous precipitate appears, and the colour changes to light green on adding more of the reagent, will give a pea-green to olive-green precipitate in 1 to 10 minutes. The colour of this then changes to reddish brown. (See *Y.B.*, 1911, 225; and also "Chemical examination of *Ænanthe crocata*" by F. Tutin, *Y.B.*, 1912, 203.)



**Cinchona Alkaloids, Relative Toxicity of.** A. C. Mac Gilchrist. (*Indian J. Med. Research*, 1914, 2, 315, 336; *Chem. Abstr.*, 1915, 9, 662.) The toxicity of the cinchona alkaloids towards guinea pigs is in the following order: cinchonidine, quinine, cinchonine and quinidine; cinchonidine being the least lethal. Quinoidine, a mixture of amorphous alkaloids, is more toxic than quinine. Differences in individual resistance of protozoa exist, not only in the same culture from day to day, but even in the same experiment on the same slide. A definite and constant relative lethality is shown to Infusoria (Ciliata) in the order, starting with the most lethal: cinchonine, quinine, quinidine, cinchonidine. *Paramecium caudatum*, though extremely sensitive to the action of these alkaloids, can live for days in solutions under a certain definite degree of concentration. The weakest solutions of the sulphate which can kill all varieties of *P. caudatum* in 20 hours are approximately: cinchonidine, 1 to 140,000; quinine, 1 to 100,000; cinchonine, 1 to 100,000; quinidine, stronger than 1 to 80,000. Ethyl-hydrocupreine and hydroquinine are less lethal than quinine to guinea pigs, but is more lethal than the other alkaloids towards *P. caudatum* and other Infusoria. When exposed for hours or days continuously to the action of fairly dilute solutions of these substances, *P. caudatum* is unable to acquire any immunity against these toxic agents. This does not preclude the possibility of immunity if the solutions were still more dilute or the exposure intermittent. This suggests the use of cinchonine in malaria.

**Colchicine, Pharmacology of.** A. Souques. (*Bull. Acad. Med.*; *Med. Review*, 1915, 18, 70.) The action of the alkaloid is discussed in reference to a case of paralysis resulting from colchicine poisoning. This was caused by the patient dosing himself with certain proprietary remedies for gout. A sudden numbness of the fingers gradually extended to the limbs and trunk, resulting in total paralysis in 12 days. It was accompanied by anaesthesia in the feet, and severe muscular pain. The loss of power persisted for about a month, then rapid improvement commenced; paralysis was but little marked three months after the attack, but muscular weakness remained. Even after 6 months the motor and sensory functions were not entirely normal. Colchicine is very slowly eliminated, and is therefore cumulative in its action. The fatal dose is variable in different persons. It is stated to be 3 centigrammes, but death may follow a dose of 3

*milligrammes*. This is largely influenced by the condition of the kidneys. The above case shows the existence of a sub-acute type of colchicum poisoning as well as the known acute form which is generally fatal. It also illustrates the care necessary in administering the drug, and for its immediate suspension on the appearance of diarrhoea.

**Copper, Therapeutic Effect of, in Tuberculosis.** H. J. Corper. (*J. Infect. Dis.*, 1914, 15, 518; *Chem. Abstr.*, 1915, 9, 106.) Copper, in the form of simple salts, as amino-acid mixture and in colloidal form, administered to rabbits by injection or by the mouth, is found to have no influence on the course of artificially induced tuberculosis.

**Corrosive Sublimate, Antidote for.** T. A. Carter. (*Drugg. Circ.*, 1914, 58, 719.) From 5 to 10 grains of sodium phosphite (not phosphate) is administered for each grain of  $\text{HgCl}_2$  taken, keeping the phosphite in excess in all cases of doubt. This salt should be stocked by pharmacists against emergency, since its prompt administration is of the utmost importance. Evacuation of the stomach should follow the administration of the antidote, a second dose of which should follow. It is claimed that 7 out of 9 cases of poisoning with  $\text{HgCl}_2$  have recovered under this treatment. The  $\text{HgCl}_2$  is reduced to  $\text{HgCl}$  by the antidote.

**Delphinium Seeds, Insecticidal Constituents of.** J. B. Williams. (*Amer. J. Pharm.*, 1914, 86, 414.) Experiments on bed-bugs show that the fixed oil and not the alkaloids is the chief active insecticide of larkspur seed. Since the drug is used entirely for external application as a parasticide, galenical preparations which contain most oil are the most efficacious. A very active fluid extract was obtained by extracting the seeds with petroleum benzin, shaking out the benzin solution with dilute  $\text{HCl}$  to remove most of the alkaloids, distilling off the solvent and dissolving the oily residue in alcohol 95 per cent. In the United States, the commercial liquid extract of larkspur seeds was found to vary greatly in alcoholic strength, the amount of alkaloid present, and the quantity of oil.

**Digitals, Tincture of Fresh Leaves of.** R. K. Robert. (*Apoth. Zeit.*, 1914, 29, 761; *J.S.C.I.*, 1915, 34, 300.) Digitalis tinctures prepared from fresh leaves with 96 per cent. alcohol are more stable than when prepared by the German official method and also more active physiologically. Such tinctures, unlike the

official tinctures, in nearly all cases had no hæmolytic action. Tinctures prepared from leaves which had been gathered for 4 days were distinctly less active than those prepared from fresh leaves. Summer leaves are preferable to October leaves. If the leaves must be dried, this should be done *in vacuo* with a rising temperature.

**Digitalis Leaves, Physiological Valuation of, and the Enzymes of Digitalis.** R a p p. (*Apoth. Zeit.*, 1914, 29, 860, 865 ; *J.S.C.I.*, 1915, 34, 449.) The physiological value of digitalis preparations can be determined accurately only by a biological process. The author prefers Hale's method to that of Focke. The guaiacum reaction is useful only as affording indications of the method of drying and preservation. In moist digitalis leaves the physiological value diminishes owing to enzyme action. The quantity of sugars formed in the digitalis plant by enzyme action is not an exact measure of the amount of digitalis glucosides lost thereby. A digitalis preparation free from enzymes may be obtained by heating the drug for 10 minutes in an autoclave at 105°C., but the secondary gastric effects produced by digitalis powder are observed with the enzyme-free preparation also.

**Digitalis, Wild and Cultivated.** J. W. H a m m e r. (*Svensk Farm. Tidsk.* ; *Chem. Abstr.*, 1915, 9, 510.) The author fails to find wild digitalis to be more active than the cultivated drug. The loss of activity of digitalis leaves does not occur so early as is generally assumed. In order to obtain definite therapeutic results preparations of the drug must be standardized by pharmacological methods. (See also *Y.B.*, 1912, 442 ; 1913, 266 ; 1914, 207, 208, 236.)

**Digitalis Leaves, Yearly Variation in Activity of.** W. L. S y m e s. (*Pharm. J.*, 1914 [4], 39, 193.) Records of yearly biological determinations of the potency of digitalis leaves from 1910-1911 to 1912-1913 are given. The first period, marked by moist, dull, variably warm weather, gave the drug of a maximum activity expressed by the factor 2.4 (standard activity being 0.8 to 1.25 as expressed by Dixon and Haynes), whereas the plants grown during 1912-1913 when the weather was drier were less than half as active, as represented by the figure 1.1.

**Distilled Water Injections, Effect of, on Bacterial Poisons.** W. J. P e n f o l d and H. V i o l l e. (*Annales Institut Pasteur*,

1914, 28, 930 ; *Chem. Abstr.*, 1915, 9, 1638.) Rabbits are sensitized to certain bacterial products by the injection of distilled water equal to one-thirtieth of the animal's weight. In the case of cholera cultures, this sensitization was observed equally with cholera organisms, and with cholera toxin from which all living organisms had been removed. In either case, the reaction is acute and immediate death may follow. This occurs equally whether the water be given first, or last ; and also follows if the toxin is injected by other than the intravenous route. A previous injection with saline solution has only a slight effect in counter-acting this action of distilled water. The same augmentation of toxicity by water injection is observed with cultures of *Proteus vulgaris*, *Bacillus pyocyaneus*, *B. dysenteriae*, *B. prodigiosus* and but feebly with tuberculin. It is not observed with HCN, alkaloids, or mineral poisons. The effect is supposed to be due to the action of the water on the red blood cells. The injection of a small quantity of hæmolyzed blood and a sub-lethal dose of cholera culture caused acute death. The lysin in the red cells seemed to be the most important factor in producing this effect. The authors propose the name "toxohæmatolysis" for this phenomenon produced by the condensation of two factors.

**Drugs and Poisons, Synergism and Antagonism of.** I. Traube and N. Onodera. (*Intern. Zeits. phys.-chem. Biol.*, 1914, 1, 133 ; *J.S.C.I.*, 1915, 34, 510.) If an alkaloid is added to the solution of the salt of another alkaloid, the extent of alteration of the surface tension depends on the relative basicity of the two alkaloids. The relative toxicity of some alkaloids is given by the following descending series of basicities : nicotine, pilocarpine, atropine, physostigmine, quinine, aconitine, veratrine. The surface tension of quinine solutions is increased by the addition of atropine, aconitine, veratrine, and nicotine. The synergetic or antagonistic influence of two substances (drugs, narcotics, etc.) on each other in the body may be due to an indirect action on the velocity of the protoplasmic reactions instead of to the direct action on the surface tension.

**Epinephrine glucosuria, Influence of Temperature on.** G. Nardelli. (*Arch. farm. sper.*, 1914, 17, 486 ; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 223.) Epinephrine glucosuria, unlike phlorhizin glucosuria, is not influenced by a rise in temperature. To produce glucosuria by a single administration of epinephrine a large dose is necessary, since a small dose may or may not

produce this effect. After a large dose, the animal retains for several months the power of responding to much smaller doses. The glucosuria lasts 6–8 hours, and in exceptional cases may persist for 24 hours. Epinephrine can also produce hæmoglobinuria.

**Ergot, Toxicological Investigation of.** F. Marino-Zuco and C. Ducciw. (*Gazz. chim. ital.*, 1914, 44, II, 437; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 900.) The EtOH in which the viscera are usually consigned is separated by filtration, rendered distinctly acid with tartaric acid and evaporated on a water-bath to eliminate most of the EtOH. The finely divided viscera and the sediment on the filter are heated for 6 hours at 75°C. with 2 volumes of 95–6 per cent. EtOH, acidified with tartaric acid, under a reflux condenser. The cold EtOH solution is filtered through a cloth and the residue pressed in the cloth. This extraction is repeated with EtOH until the latter no longer becomes coloured. The combined solutions are then evaporated on a water-bath to a small volume. This concentrated solution is mixed with the residue from the EtOH originally present and the remaining EtOH removed at a low temperature *in vacuo*. The red-brown turbid syrup obtained is dissolved in water (soln. 1) and the solution agitated for a long time with 2 volumes of Et<sub>2</sub>O in a large separator. This is repeated with fresh Et<sub>2</sub>O until it no longer becomes coloured. The Et<sub>2</sub>O dissolves red colouring matter and a small amount of fat, while the tartrates of alkaloids remain in the aqueous solution. The combined Et<sub>2</sub>O extracts are concentrated to a small volume on a water-bath at as low a temperature as possible and the residual liquid filtered if necessary and vigorously agitated with a cold saturated solution of NaHCO<sub>3</sub> in a separator. After a long time the alkaline layer separates and shows a red-yellow colour with faint violet tint if a small amount or a distinct violet-red colour if a large amount of ergot is present. This colour reaction may be masked by extraneous colouring matters and the extraction with alkali is repeated several times. The total extract is finally carefully acidified with concentrated HCl and extracted with Et<sub>2</sub>O. The Et<sub>2</sub>O solutions, which will be more or less orange-yellow in colour, are concentrated to a small volume and the residual liquid examined spectroscopically. When the red colouring matter of ergot is present 3 absorption bands,  $\lambda$ 538, 499 and 467, are present. Before making the spectroscopic examination it is advisable to repeat the NaHCO<sub>3</sub> extraction,

acidification and  $\text{Et}_2\text{O}$  extraction at least 5 or 6 times in order to obtain a sufficiently pure  $\text{Et}_2\text{O}$  solution of the colouring matter. The aqueous liquid acid with tartaric acid (soln. 1) remaining after the extraction with  $\text{Et}_2\text{O}$  was made alkaline with saturated  $\text{NaHCO}_3$  solution and the liberated alkaloids extracted with  $\text{Et}_2\text{O}$ . The faintly yellow  $\text{Et}_2\text{O}$  solution is concentrated at a low temperature; the residue is washed with a little  $\text{H}_2\text{O}$  in which ergotinine is insoluble. The ergotinine is then either purified by Tanret's method (*J. pharm. chim.*, 1878, 28, 182) or converted into the citrate by shaking with  $\text{Et}_2\text{O}$  solution several times with citric acid solution. The citrate solution is made alkaline by adding  $\text{Na}_2\text{CO}_3$  and the ergotinine extracted with  $\text{Et}_2\text{O}$ . Keller's reaction for ergotinine is then applied; the residue left on evaporating the  $\text{Et}_2\text{O}$  slowly is heated slightly with 2-3 c.c. 0.1 per cent.  $\text{FeCl}_3$  in glacial  $\text{AcOH}$  on the water-bath. The cold liquid, filtered if necessary, is placed in a test tube and 2-3 c.c. concentrated  $\text{H}_2\text{SO}_4$  added slowly. If ergotine is present a blue ring appears at the surface of separation of the two liquids and the upper layer becomes violet. If Keller's reaction fails, the  $\text{Et}_2\text{O}$  solution which was extracted with citric acid should be evaporated and the residue tested in the same way. The above method gives reliable results if the amount of ergot present is at least 1 Gm. and if the examination of the viscera is carried out not later than 7 days after burial. Advanced putrefaction of the viscera alters the colouring matter so profoundly that recognition is impeded or prevented.

**Ethane Tetrachloride is Toxic.** W. H. Willcox, T. M. Legge and B. Spilsbury. (*Lancet*, 1914, 187, 1489.) Ethane tetrachloride, employed as a rubber solvent in conjunction with benzene, acetone and methylated spirit, gives off a heavy vapour, the prolonged inhalation of which has occasioned fatal hepatitis. Where this liquid is used as a volatile solvent, means should be taken to remove its heavy vapour from the ground level of the drying-room.

**Ether-Sterilized Vaccine against Cholera.** H. Vincent. (*Comptes rend.*, 1915, 160, 378.) The author has previously shown that aqueous emulsions of the organisms of typhoid, paratyphoid, Malta fever, and also staphylococci, are sterilized by agitation with  $\text{Et}_2\text{O}$ ; and that the aqueous liquid after this treatment affords a valuable protective vaccine against the respective diseases. The cholera vibrio is now shown to be

rapidly killed by contact with  $\text{Et}_2\text{O}$ . Emulsions of this organism when shaken up with  $\text{Et}_2\text{O}$ , are completely sterilized. The separated aqueous portion, which contains the dead vibrios in a disintegrated and dissociated condition, affords an effective immunizing vaccine against cholera, the injection of which causes a prompt formation of antibodies. This last factor is one of great importance, in view of the brief period of incubation of the disease. Experiments on animals with the  $\text{Et}_2\text{O}$  sterilized vaccine have given most satisfactory results.  $\text{Et}_2\text{O}$  has a specially favourable action, since besides sterilizing the emulsion, it removes lipoids, some of which may be toxic.

**Gold Chloride and Colloidal Gold, Different Pharmacological Action of.** H. Busquet. (*Comptes rend.*, 1915, 160, 404.) The action of  $\text{AuCl}_3$  is totally different from that of colloidal Au when these are injected, in equal doses, into the dog or rabbit.  $\text{AuCl}_3$  is extremely toxic. It increases the frequency of the heart beats, decreases their volume, and occasions a fall in the blood pressure, so that immediate death may result. Colloidal gold is practically non-toxic. It lessens the frequency of the heart beats, considerably augments their amplitude, and increases the arterial pressure.

**Goldfish as Test Animals for Pharmacological Experiments.** P. S. Pittenger and C. E. Vanderkleed. (*Drugg. Circ.*, 1915, 59, 6.) The Goldfish, *Carassius auratus*, is recommended as a test animal for standardizing cardiac remedies is suggested. The method consists of simply placing the animals in varying dilutions of the drug and noting the minimum dilution which will cause death in a given time. A series of preliminary experiments are quoted as leading to the following conclusions: Gold fish are sensitive to variations of  $2\frac{1}{2}$  per cent. in the strength of the dilutions of digitalis in which they are placed. Variations due to differences in the rate of absorption appear to be practically eliminated by the use of these animals. Decreasing the strength of the dilution, increases the sensitiveness of the test. The weight of the fish may be disregarded when making tests by this method. Variations in the temperature markedly influence the resistance of goldfish to digitalis poisoning. The individual variation in the susceptibility of goldfish is much less than that found in guinea pigs and frogs. The goldfish method is unquestionably the simplest method so far proposed, and can easily be carried out by those not especially skilled in the pharma-

codynamic art. The inexpensiveness of the assay is decidedly in its favour. A sufficient number of animals can be procured at all seasons of the year.

[The use of goldfish, or other species of freshwater fishes, for this purpose has been made before. The extreme sensitiveness of fish to the toxic influence of the saponins is well known. The greater number of the fish poisons used by primitive races contain and owe their remarkable efficacy to saponin glucosides.—Ed. Y.B.]

**Holarrhena congolensis, Action of the Alkaloids of, and also of Oxyconessine.** J. H. BURN. (*J. Pharmacol.*, 1915, 6, 305–21; *Chem. Abstr.*, 1915, 9, 659). Conessine and holarrhenine have a narcotic effect in frogs, which is inappreciable in mammals. Locally these bases are anæsthetic to the rabbit eye. Necrosis is produced on local injection. Depressant effects are produced in the circulatory system due to action on smooth muscle. Oxyconessine has practically opposite effects.

**Iodine, Germicidal Value of.** T. MABEN and J. S. WHITE. (*Chem. & Drugg.*, 1915, 86, 144.) The bactericidal value of I in either EtOH or aqueous solutions is at least four times as great as that of phenol on the naked organism, such as *B. typhosus*. The authors' experiments do not show that the EtOH solution of I has any marked superiority as a bactericide over the aqueous solution. But the presence of EtOH does not, as stated by Godlee, diminish the bactericidal activity of the I. Experiments are quoted which prove these and other points concerning which there has been much controversy.

**Isostrychnine, Pharmacodynamic Action of.** B. WIKI. (*L'Union Pharm.*, 1914, 55, 341.) Isostrychnine,  $C_{21}H_{22}N_2O_8$ , is 30 to 35 times less toxic for rabbits than strychnine; its toxicity approximates to that of brucine. Its action on the nervous system resembles more closely that of curare than of strychnine; doses which kill by arresting respiration do not affect the cardio-vascular system. Its tetanizing action is slight, and only seen under certain conditions. Pharmacologically, isostrychnine is placed between curare and brucine.

**Lead Poisoning from the Use of Cosmetics.** G. W. ROBINSON. (*J. Am. Med. Assoc.*, 1915, 64, 814–5; *Chem. Abstr.*, 1915, 9, 1204.) Reports are given of two cases of lead poisoning resulting



from the use of a cosmetic called Flake White, which is a levigated lead subcarbonate and is applied to the face with a moist sponge.

**Loco-weed Disease of Sheep, A Summary of Studies of.** H. T. Marshall. (*Bull. Johns Hopkins Hosp.*, 1914, 25, 234-6; *Chem. Abstr.*, 1915, 9, 653.) The author doubts the existence of bona fide loco-weed poisoning and believes that the losses attributed to loco-weed disease are due to other causes which can be usually ascertained by careful study. All of the severely "locoed" sheep examined were suffering not from locoism, but from underfeeding combined with parasitic infection.

**Manganese, Non-poisonous Properties of.** T. Bokorny. (*Chem. Ztg.*, 1915, 38, 1290; *Chem. Abstr.*, 1915, 9, 1510.) In distinction to the other heavy metals, Mn is non-poisonous if it is in the form of its salts. When yeast is added to a 1 per cent.  $\text{MnSO}_4$  solution budding was observed, although in a 3 per cent. or 5 per cent. solution no action took place. No budding took place in similar solutions of Fe and Ni salts. Yeast steeped for 24 hours with  $\text{MnSO}_4$  showed no test for Mn with the usual reagents after washing thoroughly; yeast similarly treated with solutions of  $\text{FeSO}_4$  and  $\text{CoSO}_4$  could not be washed free from the respective metals. The innocuous character of Mn salts is attributed to the fact that, unlike the other heavy metals, Mn does not combine chemically with the protoplasm.

**Maté Tea : A Comparative Study of Stimulants.** R. Brieger. (*Pharm. Zentralhalle*, 1914, 55, 975; *Chem. Abstr.*, 1915, 9, 1640.) Maté tea is prepared from the leaves of *Ilex paraguayensis* and several other species of *Ilex*, indigenous to S. America. It yields a pleasant beverage that is considerably less astringent than ordinary tea. One sample of the leaves yielded 2.5 per cent. of caffeine.

**Mercuric Chloride, Alkaloidal Antidote for.** W. A. Hall. (*J. Amer. Pharm. Assoc.*, 1915, 4, 183.) Remove the stomach contents as thoroughly as possible, give plenty white of eggs and remove in the best way, wash out the stomach thoroughly, then for every two grains of  $\text{HgCl}_2$  supposed to have been taken, administer the following: Potassium iodide, 7.35 grains; quinine hydrochloride, 4 grains; dissolved in water, 4 ounces. For 10 grains of  $\text{HgCl}_2$ : Potassium iodide, 36.75 grains; quinine hydrochloride, 20 grains; distilled water, 4 ounces; hydro-

chloric acid (10 per cent.), m. 45. It forms a precipitate with the HCl, insoluble in dilute acids or alkali carbonates (0.2 per cent.). A solution could be kept on hand ready for use of the formula above, with the addition of HCl to make it 0.2 per cent.

**Mercuric Chloride Poisoning in Animals treated Unsuccessfully by Administration of Hall's New Antidote.** H. G. Barbour. (*J. Amer. Med. Assoc.*, 1915, **64**, 736; *Chem. Abstr.*, 1915, **9**, 1204.) Experiments with mice and rabbits showed the antidote to be of no value. While it precipitates Hg from saline solutions in dilutions of 1 : 25,000 it fails to precipitate Hg from solutions of ox serum in dilutions of 1 : 1000, showing that Hg forms a firmer combination with the body tissues or blood serum than with the antidote.

**Mercury Organic Compounds, Relationship of Chemical Constitution to Toxic and Curative Powers of.** W. Schoeller and W. Schrauth. (*Chem. Ztg.*, **36**, 1112; *Chem. Abstr. Amer. Chem. Soc.*, 1914, **8**, 3098.) The toxicity of various Hg compounds bears a close relationship to their stability. The extraordinary difference in toxicity between the highly poisonous Hg-diethyl and the absolutely harmless Hg dipropionic acid led the authors to consider another important factor, speed of elimination. Toxicity appears to equal stability divided by speed of elimination. The same conclusions apply to the As preparations of Ehrlich.

**Opium, Constipating Constituents of.** Makoto Takahashi. (*Arch. Physiol.*, **159**, 327; *Chem. Abstr.*, 1915, **9**, 486.) A combination of morphine with codeine produced a marked increase of the constipating action on colocynth diarrhoea in cats. Even by the combination of  $\frac{1}{4}$  of the smallest active dose of morphine with  $\frac{1}{40}$  to  $\frac{1}{400}$  of that of codeine a considerable constipating action was found. This combination does not increase the action of morphine upon the central nervous system. In normal cats a noticeable increase of the action of morphine upon the intestine may be produced by codeine, but a much larger dose is needed than is the case in colocynth cats. The evacuation of the stomach is retarded by the doses of codeine-morphine that act upon the normal intestine, but not by the small doses that stop colocynth diarrhoea. In opium and pantopon, morphine and codeine are not present in the proportions necessary for constricting action. With equal

amounts of morphine, a dose of morphine-codeine has a stronger constricting action than pantopon or opium tincture. The action of pantopon is stronger than that of morphine but weaker than that of opium tincture. Opium and pantopon contain substances which decrease the constipating action of morphine-codeine. Besides morphine and codeine there is probably no other alkaloid present in opium that increases the constipating action to any measurable degree. The influence of the so-called "alkaloid residue" is very small. Meconic acid has no effect. It is possible that substances are present in opium which increase this action to a slight degree. It is possible that the action of opium preparations upon men may be due to the same cause.

**Phenol Poisoning, Washing out the Stomach with  $\text{Na}_2\text{SO}_4$  for.** D. I. M a c h t. (*Bull. Johns Hopkins Hosp.*, 1915, 26, 98-104; *Chem. Abstr.*, 1915, 9, 1508.) Liquid carbolic acid was administered to dogs and cats by means of a stomach tube, followed by lavage at different periods of time after the appearance of toxic symptoms. Plain water, aqueous solutions of EtOH and concentrated solutions of  $\text{Na}_2\text{SO}_4$  were used for washing out the phenol. The chances of recovery depend on the amount of phenol swallowed, on the promptness with which lavage is begun and on the solution used for lavage. Food in the stomach greatly increases the chances of recovery. A strong solution of  $\text{Na}_2\text{SO}_4$  is the most efficient agent, recovery being effected in one dog by lavage with such a solution 45 minutes after the first dose of phenol and after a dose of 22 c.c. had been given. The action of the  $\text{Na}_2\text{SO}_4$  is not a chemical one but is probably due to the hindrance of absorption and possibly also to purgation. Administration of EtOH after poisoning aggravates the symptoms and hastens death; but an animal intoxicated with EtOH withstands better the effects of phenol taken afterwards. *The use of alcohol in carbolic-acid poisoning should therefore be strongly discouraged.* If a solution of  $\text{Na}_2\text{SO}_4$  is not available, water is the most efficient medium to employ. Experiments were also made using egg albumin,  $\text{Na}_2\text{SO}_3$ , lime water and saccharated lime solution, but none of them proved satisfactory antidotes when the saline solutions were not used as washes.

**Pituitary Extract Administered before Surgical Operation.** H. K a h n and L. E. G o r d o n. (*J. Amer. Med. Assoc.*, 1914 64, 301.) The administration of pituitary extract for

hypodermic injection, in doses of 12 minims for children or 15 minims for adults, 15 minutes before giving the anæsthetic is of decided value in increasing the coagulability of the blood. The hæmorrhage in nose and throat operations is therefore much reduced.

**Potassium and Sodium Iodides and of the Iodine Ion, Action of, on the Heart and Blood Vessels.** D A V I D I. M A C H T. (*Bull. Johns Hopkins Hosp.*, 1914, 25, 278-84; *Chem. Abstr.*, 1915, 9, 659. Cf. *C.A.* 8, 3694.) Experiments were performed with normal Locke solution modified so as to contain the substance, the action of which it was desired to test. These solutions were used in perfusing the blood vessels of cold- and warm-blooded animals, in perfusing amphibian and mammalian hearts, in studying the action of the substances on arterial strips, and in blood pressure experiments on dogs. The K ion produced relaxation of the blood vessels and pronounced depression of the heart; on the other hand the Na ion slightly stimulated both. I ion in isolated organs is a powerful stimulant, but in intact animals the stimulating effect is greatly inhibited by chemical combination of the I with the blood proteins. NaI produces no depressing effect, since both the Na and I ions are vascular constrictors and cardiac stimulants; KI on the other hand exhibits the depressing effect of the K ion to a marked degree. In cases of aneurism, therefore, it is not a matter of indifference which iodide is chosen for depressing the circulation. The effect of KI in lowering blood pressure is due entirely to the K, and the author considers that this can be more efficiently attained by the use of other K salts.

**Sanatogen, Feeding Value of, Compared with Commercial Casein with Respect to Maintenance and Growth.** J. P. S T R E E T. (*J. Amer. Med. Assoc.*, 63, 1831; *Chem. Abstr.*, 1915, 9, 124.) Sanatogen costing \$3.12 per lb. (wholesale) was compared with compound casein costing \$0.10 per lb. On a water-free basis sanatogen consists of about 90 per cent. of casein, 5 per cent. of Na glycerophosphate, with a small amount of an unidentified N compound containing S and P and a small amount of P in inorganic combination. A comparative feeding of 6 rats during 9 weeks showed no nutritive superiority of sanatogen over commercial casein. A comparative feeding of 4 rats during 11 weeks showed slightly greater but insignificant increase in weight for sanatogen over casein. In a ration in which artificial had

been substituted for natural protein-free milk, sanatogen showed no advantage over casein in checking the loss in weight.

**Sodium instead of Potassium Salts, Substitution of, for Medicinal use.** E. White. (*Pharm. J.*, 1914 [4], 39, 286.) In the present shortage of K salts, in consequence of the war, the desirability of substituting Na salts in prescribing, for those of K is pointed out. The former are readily obtainable in quantity, and are at least as effective as the latter.

**Sodium Salicylate, Intolerance of, Caused by Traces of Free Salicylic Acid.** R. Lecoq. (*Bull. Sci. pharm.*, 1915, 22, 84.) A number of cases of intolerance observed with a certain lot of sodium salicylate, in private and hospital practice among juvenile and adult patients, enabled the author to trace the cause to a trace of free salicylic acid in the consignment. To detect this an aqueous solution was shaken out with  $\text{Et}_2\text{O}$ . After washing, the  $\text{Et}_2\text{O}$  was separated and evaporated. The residue gave a positive reaction for salicylic acid. The amount of free acid was determined by titrating the aqueous solution of the salt with N/10 NaOH with phenolphthalein indicator. It amounted to only 0.069 per cent. That this small amount of free acid was the cause of the trouble was proved by the fact that when it was neutralized with  $\text{NaHCO}_3$  no intolerance was developed, even by children who had been adversely affected by the faintly acid salt. The aqueous solution of the original salt was very feebly acid to litmus, but gave a distinctly acid reaction to phenolphthalein solution, previously reddened with a minute trace of alkali.

**Squill, Physiological Action of.** W. K o p a c z e w s k i. (*Biochem. Z.*, 66, 501; *Chem. Abstr.*, 1914, 8, 3824.) Scillitin is a poison, which is toxic in doses of 1 Mgm. per kilo body-weight; scillidiuretin is a diuretic, which causes the production of twice the normal amount of urine in rabbits. Extract of squill is more poisonous for guinea pigs and rabbits than is scillitin. (See also *Y.B.*, 1914, 100.)

**Strophanthin in the Digestive Tract, Behaviour of.** F. J o h a n n e s s o h n. (*Arch. Exp. Path. Pharm.*, 1914, 78, 83; *Chem. Abstr.*, 1915, 9, 663.) The decreased poisonous effect of strophanthin in the digestive tract is not due to the action of enzymes but of the H and HO ions. The HO ions<sup>1</sup> weaken only crystalline k-strophanthin, while the H ions act upon the crystalline and

amorphous *kombé*-strophanthin as well as *Tinctura Strophanthi*. There is no decrease in the activity of *gratus*-strophanthin. Many negative results of strophanthin therapy can be traced in part to the effect of acid digestion products. For this reason the crystalline *g*-strophanthin should be used internally, as this is resistant to the digestive juices.

**Strophanthin, the Identification of, in the Stomach and Intestine.**  
F. J o h a n n e s s o h n. (*Archiv. Exper. Path. Pharm.*, 1914, **78**, 92; *Chem. Abstr.*, 1915, **9**.) The slight activity of strophanthin after poral administration as compared with intravenous injection is partly due to slow resorption (*g*-strophanthin), partly to the change by the digestive juices. Amorphous *k*-strophanthin is changed by gastric HCl largely into strophanthidin, but to a slight extent into a completely inactive resinous product. Amorphous *k*- and crystalline *g*-strophanthin may be biologically identified in the stomach and intestinal contents of rabbits, poisoned with sufficiently large doses; also by the use of the  $H_2SO_4$  reaction. This could not be made quantitative.

**Strychnine, Chemical Constitution and Physiological Action of.**  
C. P a d e r i. (*Arch. farm. sper.*, 1914, **18**, 66-87; *Chem. Abstr. Amer. Chem. Soc.*, 1915, **9**, 227.) The convulsive action of strychnine is not due to the group  $=N-CO-$ , since piperidone and pyrrolidone, which contain this group, do not produce convulsions; and since the conversion of strychnine into strychnol, whereby the group  $=N-CO-$  is eliminated, does not diminish the convulsive action.

**Taste and Chemical Constitution of some Organic Substances.**  
G. C o h n. (*Pharm. Zentralhalle*, 1914, **55**, 735; *Chem. Abstr.*, 1915, **9**, 464.) The structure of a number of oxime-acetic and ketonic acids is discussed with reference to their influence on the gustatory nerves. The groups  $-CH:NOCH_2CO_2H$  and  $=C:NOCH_2CO_2H$  attached to a  $C_6H_5$  nucleus uniformly give sweet compounds. The presence of an  $NO_2$  radicle tends to give a bitter taste which is so pronounced, when in the para-position to the "sweet" group, as to overcome the sweet taste. The Na salts of benzoylbenzoic acids are seldom purely bitter or sweet, but often are bitter-sweet or sweet-bitter. They are more sweet if a  $CH_3$ ,  $CO_2H$ ,  $OH$ ,  $OCH_3$ , or  $N(CH_3)_2$  radicle is in the para-position to the CO group of the benzoyl

complex. When these radicles are in the ortho-position to the CO group the taste is generally bitter. Isomerism sometimes has an influence upon taste, e.g., the Na salt of  $\alpha$ -methoxynaphthoyl-o-benzoic acid is very bitter, while the  $\beta$ -compound is strongly and purely sweet.

# PHARMACY

## DISPENSING

**Acetanilide, Insolubility of, in Prescription.** (*Drugg. Circ.*, 1915, 59, 374.) Acetanilide, 70 grains; citrated caffeine, 15 grains; codeine sulphate, 6 grains; citric acid, 4 grains; sodium bromide, 160 grains; Hollands gin, 2 drachms; alcohol, 6 drachms; tincture of gelsemium, 2 drachms; cinnamon water, to make 4 fl. oz. The acetanilide is soluble in the alcoholic ingredients, but is precipitated on adding the water. It had been dispensed previously as a clear mixture. This can be obtained only by materially increasing the alcoholic strength of the vehicle, which should not be done without the sanction of the prescriber.

**Benzene Capsules for Intestinal Medication.** R. A. Kuever. (*Drugg. Circ.*, 1915, 59, 289.) Capsules containing  $C_6H_6$  are now largely used in hospital practice for the treatment of leukaemia. It is very important that these should be quite unacted on by the gastric secretion, so that the contents are not liberated in the stomach. If this occurs, vomiting will ensue. The capsules should therefore be coated with keratin in acetic acid, and with salol. The benzene should be pure "crystallizable"  $C_6H_6$ . A mixture of this with equal volumes of olive oil is generally used for filling the capsules. Only hard capsules should be used, and when filled these should be stored in a cool place. Warm water is used to seal the cap of the capsule. This must be done perfectly. After coating, one of the capsules should be tested by immersing it for 15 minutes in a 1:500 solution of HCl at 37.5°C. with occasional agitation. It should show no sign of disintegration. The following table shows the capacity of various capsules for the benzene oil mixture and the amount of salol required to cover them.



No. of Capsule.	Capacity.	Salol Required.
000	25 minims	5 grains
00	16 "	4 "
0	12 "	3 "
1	8 "	2½ "
2	6 "	2 "
3	4 "	2 "
4	1½ "	1½ "
5	1 minim	1 grain

**Capsules of Apiol with Powders.** (*Drugg. Circ.*, 1915, 59, 374.) The following was dispensed by placing the solid ingredients in the capsule proper and dropping the apiol into the cover or cap and then sealing the two parts together: Exsiccated ferrous sulphate, 1 drachm; potassium carbonate, 1 drachm; arsenious acid, 1 grain; extract of nux vomica, 8 grains; apiol, 1½ drachms. Mix and divide into 40 capsules. This may be compounded satisfactorily by adding 20 grains of starch to the apiol contained in a mortar, and after the apiol had been absorbed adding the other ingredients. A powder which can easily be encapsulated is thus obtained. Magnesium carbonate might be used in place of starch. The quantity of absorbent powder to be used must be determined by experiment. A good method of procedure is to weigh out more of the absorbent than is to be used and to then add it in small portions until the desired result is obtained. The remaining powder is then weighed, and by subtracting this from the amount originally weighed, the amount that has been used is obtained. This can be noted on the prescription for future reference.

Another method would be to make a pill mass of the apiol with some suitable excipient, divide it into 40 equal parts and place each separate pill into the body of a capsule, after which the other powders, previously mixed, can be added in the usual manner. Dropping the apiol is not a sufficiently accurate method of dispensing this.

**Creosote Pills.** J. Schirmer. (*Pharm. Zeit.*; *Nat. Drugg.*, 1914, 44, 294.) Creosote, 10 Gm.; glycerin, 1 Gm.; licorice powder, 19 Gm.; mucilage of acacia, 4 Gm. Make 200 pills.

**Dispensing Camphor.** J. L. Lascoff. (*Drugg. Circ.*, 1914, 59, 613.) Camphor in pill or capsule form should always be put up in bottles, and not in boxes. The pills, or tablets,

should be coated, to prevent evaporation. *Camphor in Pills*.—(1) Camphor, 2·0 Gm. Mix and make 30 pills. The camphor is triturated with about 1 Gm. of powdered soap, and a few drops of castor oil are added. This makes a mass which does not harden and should be dispensed in a glass container well corked. (2) Camphor, 20 grains; menthol, 10 grains. Mix and make 25 pills. Triturate the camphor and menthol. To the resulting liquid, add a mixture of extract of licorice and powdered licorice root, and then add a few drops of water. This makes a very good mass. The finished pills should be coated to prevent evaporation of the camphor. *Camphor in a Mixture*.—Ammonium chloride, 4·0 Gm.; powdered camphor, 0·6 Gm.; syrup of wild cherry, 30·0 Gm.; distilled water, enough to make 120·0 Gm. Triturate the camphor with 1 Gm. of granulated acacia to a fine powder, and then add the water and syrup little by little.

**Dispensing Ext. Filicis Liq.** H. B. Sh a r m a n. (*Pharm. J.*, 1915 [4], 40, 789.) Pour the required quantity of the extract into a measure, add to it Tinct. Quillaïæ, q.s., and mix vigorously with a glass rod until emulsified; then add a little water, transfer to bottle, and add remainder of water and the flavouring agent, shaking after each addition. The result is a perfect emulsion, containing the exact quantity of extract, and no oil is left on the side of the measure, so that it is easily cleaned.

**Gelatin Capsules, Treated with Formaldehyde Solution, for Ipecacuanha Medication.** E. L. De L a n n e y. (*J. Am., Med. Assoc.*, 1914, 63, 1506; *Chem. Abstr.*, 1915, 9, 123.) Capsules soaked in a 20 per cent. solution of HCHO for a few hours and dried became very brittle and somewhat distorted, but not so as to prevent their subsequent filling. This treatment renders the capsules insoluble in acid media and furnishes a satisfactory method for the administration of ipecacuanha. The capsules pass through the stomach, and do not liberate the drug until in the intestine. Nausea and emesis are thus avoided.

**Guaiacol Carbonate in Pills.** (*Chem. & Drugg.*, 1915, 86, 403.) To make three dozen small pills of guaiacol carbonate containing 2 grains in each, the guaiacol carbonate should be triturated with 3 grains of powdered tragacanth and massed with 26 grains of syrup of glucose. This gives a pill under 3 grains in weight which does not become hard quickly.

**Incompatibles.** W. L. Wilbur. (*J. Amer. Pharm. Assoc.*, 1915, 4, 590.) *Quinine with Organic Acids; Formation of Quinotoxin.*—The change of colour noted in liquid galenical preparations containing quinine and iron citrate in the form of elixirs and syrups and other combinations of quinine in presence of acetic, tartaric, citric, malic, lactic or formic acids, is stated to be due to the formation of quinotoxin. Such preparations are known to darken on storage, and the presence of ferric salts is stated to accelerate the change. A preparation so darkened is not only unsightly, it may be even poisonous. It is considered probable that some cases of "idiosyncrasy to quinine" are due to the presence of quinotoxin thus formed. Cinchotoxin, formed under like conditions from cinchonine, is much more poisonous than quinotoxin. Such preparations of quinine and acids should be stored in amber bottles and should not be dispensed when discoloured. The change of quinine to quinotoxin by organic acids proceeds much more rapidly when they are heated. Consequently such mixtures should not be exposed to a high temperature. A small amount of mineral acid will hinder or prevent the change. An instance of a similar action has been observed between quinine and aspirin. When mixed and enclosed in capsules, these ingredients became liquid and dark coloured in a year. The liquid had the odour of acetic acid. Probably the free acids had partly converted the quinine into quinotoxin.

*Ferric Salts and Organic Acids in Light.*—Effervescence, at first attributed to fermentation, in a specimen of malt beef and iron wine, containing 20 per cent. of EtOH, first directed attention to this. The wine contained ferric citrate or tincture of iron citrochloride, as well as a citrate. The effervescence was traced to the total decomposition of the citric acid, into  $\text{CO}_2$  and water by the action of sunlight on the mixture, the Fe being reduced to the ferrous state. Amber bottles inhibit this reaction which is entirely photochemical, and many ferric preparations are liable to it in presence of organic acids.

*Boric and Tartaric Acids in Tablets.*—Tablets containing  $\text{HgCl}_2$ ,  $\text{AmCl}$ , tartaric and boric acid do not keep well. An odour of free HCl is evident. The two acids unite to form borotartaric acid, which is stated to be the cause of the liberation of the HCl from  $\text{AmCl}$ .

**Incompatible Dressings of Tincture of Iodine and Mercuric**

**Chloride.** L. Reutter. (*Bull. Sci. pharm.*, 1914, 463; *Schweiz. Apoth. Zeit.*, 1915, 53, 107.) Sublimate dressings should not be applied to wounds previously treated with tincture of iodine, or irritant  $\text{HgI}_2$  will be formed on the wound surface. Should this have occurred through inadvertence to this obvious incompatibility, the harmful  $\text{HgI}_2$  should be removed by washing with a solution of KI, 1 : 10.

**Iodine and Ichthyol, Incompatible Prescription of.** (*Drugg. Circ.*, 1915, 59, 172.) Tincture of iodine, 6 drachms; ichthyol, 6 drachms; olive oil, 6 drachms; powdered acacia, 2 drachms; distilled water, enough to make 3 ounces. A satisfactory emulsion may be obtained by dissolving the ichthyol in the oil and emulsifying the mixture with the acacia and water in the usual manner. When the tincture of iodine is added to the finished emulsion, the acacia is not precipitated, but the mixture assumes a bluish-green tint, due to a reaction between iodine and ichthyol.

**Isotonic Collyria.** G. Siboni. (*Boll. Chim. farm.*, 1914, 54; *J. Pharm. Chim.*, 1915, 11, 241.) The tears which normally bathe the corneo-conjunctival epithelium have the congealing point  $-0.86^\circ\text{C}$ ., which is the freezing point of a solution of 14 Gm. of NaCl in 1,000 c.c. of water, or of a solution of a similar molecular concentration. In preparing collyria, therefore, the amount of NaCl used must be reduced in proportion to the quantity of other substance introduced, in order to keep the solution isotonic. For instance, the molecular weight of eserine salicylate being 413 and that of NaCl 58.5, a collyrium containing 5 in 1,000 of eserine salicylate isotonic to the lachrymal secretion is prepared by deducting  $\frac{5 \times 58.5}{413}$

$=0.7$  of NaCl, from 14, the normal amount of NaCl;  $14 - 0.7 = 13.3$  Gm., which is the weight of NaCl used with 0.5 Gm. of eserine sulphate to produce 1,000 c.c. of collyrium. The following are the proportions of the salts named requisite to produce isotonic collyria: (1) Crystalline zinc sulphate, 10; crystalline sodium sulphate, 27. (2) Crystalline cadmium sulphate, 10; crystalline sodium sulphate, 27. (3) Crystalline zinc sulphate, 5; crystalline cadmium sulphate, 5; crystalline sodium sulphate, 27. (4) Crystalline copper sulphate, 10; crystalline sodium sulphate, 25. (5) Crystalline silver nitrate, 10; crystalline sodium nitrate, 15.52.

**Isotonic Eye Lotions.** (*Prescriber*, 1915, 9, 125.) An eye lotion, to yield the best results, should be isotonic with the tears. Such tonicity is represented by a solution of NaCl of 1.4 per cent. A 1 per cent. solution of cocaine hydrochloride should contain 1.25 per cent. of NaCl.  $\text{ZnSO}_4$  requires 0.027 per cent. of  $\text{Na}_2\text{SO}_4$  to a 1 per cent. solution, and  $\text{AgNO}_3$  needs 0.01552 per cent. of  $\text{NaNO}_3$  for proper isotonicity. The following prescriptions are recommended: (1) R Cocain. hydrochlor., 0.5 Gm.; sodii chloridi, 0.625 Gm.; aq. destil., to 50.0 c.c. (2) R Zinci sulphat., 0.5 Gm.; sodii sulphat., 1.35 Gm.; aq. destil., to 50.0 c.c. (3) R Argenti nitrat., 0.5 Gm.; sodii nitrat., 0.766 Gm.; aq. destil., to 50.0 c.c.. A 2 per cent. solution of boric acid (i.e., half the strength of a saturated solution) requires no addition, being practically isotonic.

**Mercuric Benzoate, Incompatibility of, with Cocaine Hydrochloride; and the Unsuitability of Mercuric Benzoate for Therapeutic Use.** R. Guyot. (*J. Pharm. Chim.*, 1915, 11, 24.) Although not infrequently prescribed in various solutions for hypodermic injections, mercuric benzoate cannot be considered a suitable salt for the purpose. Its solubility is not great. It is readily decomposed. In aqueous solution when heated it is dissociated into  $\text{HgO}$  and  $\text{C}_6\text{H}_5\text{COOH}$ . When prescribed with NaCl in solution either  $\text{HgCl}_2$  is formed if the benzoate is pure or  $\text{HgCl}$  if mercurous salt is present, as most frequently occurs. When cocaine hydrochloride is prescribed with mercuric benzoate solution to lessen the pain of injection, practically all the alkaloid is precipitated. The author would dismiss mercuric benzoate from therapeutic use.

**Ointments, Reform in the Dispensing of.** F. W. Nitary. (*J. Amer. Pharm. Assoc.*, 1915, 4, 147.) The use of collapsible tubes for sending out ointments is advocated. These are cleaner, preserve the ointment from oxidation and accidental contamination, are more convenient in use, and have a better appearance than pots or other vessels. The tubes are also cheaper. They may be made attractive by lacquer and packed in suitable boxes. The filling of individual tubes is most easily accomplished by rolling the preparation into cylindrical form with a piece of paper, slipping this into the tube, flattening the paper cylinder just beyond the portion holding the ointment and then withdrawing the paper, continuing to flatten it as it is being withdrawn, so as to force the ointment out of the paper into

the tube. After one trial the operation will be found just as quick and simple as filling a jar with a spatula. When tubes are used for such preparations as should not come in contact with metals, the tube must, of course, be previously coated with a suitable lacquer. Collodion or an ethereal tincture of tolu are suitable for this purpose. A little of this is poured into the tube and rotated so as to completely cover the inner surface, the surplus is then poured out and the tube allowed to dry. The operation takes only a few minutes.

**Peruvian Balsam Emulsion.** (*Nat. Drugg.*, 1915, 49, 68.) The following prescription has been presented: R Emuls. bals. peruv., 5 per cent., ℥viii. M. s.a. To be applied to the burn. A small quantity of powdered tragacanth in a dry mortar is mixed with a little alcohol, and sufficient water is added to form a stiff paste. The Peruvian balsam is then intimately mixed with this; finally, sufficient water is added, gradually, with constant trituration, to bring the emulsion to the prescribed volume.

**Phenol, Solubility in Paraffins.** G. Barker. (*Pharm. J.*, 1914 [4], 39, 721.) Phenol is almost insoluble in paraffins. It may be expected, therefore, to separate out in Ung. Acid. Carbolic, B.P., 1914. When phenol, 13 grains, is dissolved in 1 fluid ounce of liquid paraffin, by warming, it crystallizes out again when the solution cools. The published statement that it is soluble in this vehicle in the proportion of 1:20 is incorrect.

**Potassium Iodide and Calcium Sulphide in Mixture.** (*Chem. & Drugg.*, 1915, 86, 754.) The query is raised as to the compatibility of the following prescription: Pot. ioidid., gr. v.; pot. bicarb., gr. v.; calcii sulphid., gr. j.; sp. ammon. ar., ℥x.; syrup. zingib., ℥j.; aquam ad, ℥ss. Ft. mist. Mitte ℥viii. ℥ss. ter in die. This may quite safely be dispensed. The calcium sulphide, being fairly light, shakes up freely and does not precipitate quickly. About a grain of tragacanth to the ounce gives it a finish.

**Potassium Iodide Solutions, Aqueous, Preservation of.** — Bouyer. (*Bull. Soc. Pharm. de Bordeaux; Répertoire de Pharm.*, 1915, 27, 143.) Although strong solutions of KI are known to keep better in the dark than when exposed to light, they are by no means stable, even under the latter conditions.

The addition of a little  $\text{Mg}(\text{OH})_2$  will render them quite permanent even in sunlight. For instance, the following solution will keep indefinitely: KI, 15 Gm.; distilled water, 300 c.c.;  $\text{Mg}(\text{OH})_2$ , 2 Gm. The KI is dissolved in a little of the water. The  $\text{Mg}(\text{OH})_2$  is suspended in the rest in another vessel and filtered into the KI solution. The faint alkalinity due to the trace of  $\text{Mg}(\text{OH})_2$  dissolved is negligible.

**Prescription Difficulties.** J. L. Lascoff. (*J. Amer. Pharm. Assoc.; Nat. Drugg.*, 1915, 45, 239.) (1) Chloretone, 1 grain; homatropine hydrochloride, 1 grain; camphor water, 2 drachms. A precipitate was formed which dissolved on warming and later re-precipitated on cooling. On using distilled water instead of the camphor water, a clear solution was obtained. After exposing the first mixture in an open vessel for a time, the solution became clear, showing that the precipitation was due to excess of camphor. The liquid being already saturated with the latter cannot dissolve any other salt which is soluble only in this proportion (chloretone is soluble 1 in 100). This can also be applied to other saturated solutions in which no other salt can be dissolved. (2) Sodium bromide, 15.0 Gm.; antipyrine, 2.0 Gm.; glycerin, 30.0 c.c.; water, q.s., to 120 c.c. This mixture may have a green colour. This is due to traces of nitrous acid present in glycerin, which reacts with the antipyrine and gives the green colour. By the use of glycerin C.P. a colourless mixture is formed. (3) Potassium citrate, 2 drachms; syrup of ipecac.,  $\frac{1}{2}$  drachm; syrup of wild cherry, 1 oz.; water, to 3 oz. The potassium citrate neutralizes the acetic acid, which is added in preparing the syrup of ipecac., to hold in solution the constituents of ipecac., as emetine and others. As the acid is neutralized, the constituents are precipitated. To prevent this, add to the syrup of ipecac. a few drops of acetic acid before mixing. In this case a clear mixture will be obtained. (4) Calamine, 4.0; zinc oxide, 6.0; sweet oil of almonds, 30.0; water, to 120.0. If this prescription is compounded in the order it is written, the oil separates, and no amount of shaking will cause it to mix. To have this prescription properly made, mix the oil with an equal amount of lime water, and shake until an emulsion is formed. Then add a few drops of fluid extract of quillaia and gradually add the remainder of the lime water and the glycerin. Mix the powders in a mortar, and rub down with the mixture. (5) Mercuric iodide, 0.2;

potassium iodide, 30·0 ; tincture of iodine, 0·4 ; syrup of ferrous iodide, 45·0 ; water, to 120·0. This prescription, which is red on first mixing, becomes decolourized on standing. The diluted hypophosphorous acid of the *syrupus ferri iodidi* (U.S.P., 1900) reacts with the free iodine of the tincture, forming hydriodic acid, which is colourless. To have the free iodine which is wanted present in this prescription, therefore, the syrup ferrous iodide (1890), which does not contain hypophosphorous acid, should be used. This forms a clear, red mixture, which remains so on standing. N.B.—The physician informed the patient not to accept the prescription unless it was of red colour.

(6) Solution of potassium arsenite, 2 drachms ; syrup of ferrous iodide, 2 oz. *Liq. potass. arsenitis* contains potass. carbonate ; the potassium bicarbonate on boiling being converted into carbonate. This—if the syrup does not contain diluted hypophosphorous acid, as the syrup of U.S.P. 1890—will react with the ferrous iodide, forming a precipitate of ferrous carbonate, which is converted into ferric hydroxide. To prevent this precipitation, use a syrup containing an accurate amount of diluted hypophosphorous acid, or neutralize the Fowler's solution before mixing, and a perfectly clear mixture is obtained.

(7) Cade oil, 20 c.c. ; water, to 120 c.c. *Oleum cadini* could not be emulsified by the ordinary emulsifying agents, neither could a suspension of the oil in water be obtained. The best way to prepare this is to emulsify the oil with the yolk of an egg, then adding a few drops of fluid extract of quillaia. This helps saponification.

**Quinine and Ammonium Carbonate in Mixture.** (*Chem. & Drugg.*, 1915, 86, 753.) The following mixture has been found difficult to dispense in a presentable form : Quin. sulph., ʒj. ; acid. sulph. arom., ʒiiss. ; liq. strychnin., ℥80 ; ammon. carb., gr. 48 ; glycerini, ʒss. ; aquam ad, ʒviij. Ft. mist. ʒss. every 5 hours. As the ammonia is largely in excess of the acid, it is obvious there will be a precipitate of quinine carbonate, which should be suspended in a state of fine division. Place the quinine sulphate and ammonium carbonate in a mortar and triturate together ; add the glycerin, and mix well ; then introduce gradually the aromatic sulphuric acid and solution of strychnine, continuing the trituration till the precipitate is uniformly smooth ; lastly, incorporate the water in successive portions to produce the eight fluid ounces required. The pre-



cipitate is in this way obtained so fine and light that it diffuses uniformly and remains in suspension long enough to permit a dose poured out to contain its proper proportion of the medication. As the mixture is wanting in "body," however, the particles of the precipitate remain on the move after the medicine has been shaken and give the whole a grey look. The appearance of the mixture is much improved by the addition of 8 grains of tragacanth, which should be triturated with the quinine and ammonia.

**Quinine and Potassium Citrate in Mixture.** (*Pharm. J.*, 1914 [4], 39, 750, 791.) Difficulty was experienced by one dispenser with the following mixture: R Quinin. sulph., grs. viii.; pot. cit., ℥iii.; syr. limonis, ℥i.; aq. chlorof., ad. ℥viii. The quinine sulphate was first rubbed down with the potassium citrate, with the result that a powdery deposit resulted in a milky-looking mixture. The second time the quinine sulphate was rubbed down with the syrup of lemon until a clear solution was formed, which was then added to the potassium citrate in solution in the bottle. This mixture, standing, deposited large crystals, although when first dispensed a clear mixture was the result. The longer it stood the larger the crystals became. Another dispenser dissolved the quinine sulphate in the syrup of lemon, then diluted with nearly 3 oz. of water, and then dissolved the potassium citrate in 4 oz. of water and mixed the two solutions together. No crystals were formed at all, after standing 24 hours.

**Reduced Iron and Acid in Prescription.** (*Mid. Drugg.*, 1915, 49, 68.) R Ferri redacti, ℥i.; tinct. cinchonae co., f℥iij.; acidi hydrochloric dil., f℥ij.; aquae destillatae, q.s. ad f℥iv.; m.sig. f℥ij., t.i.d. The reduced iron and the acid should be gently heated [with a little of the water] until evolution of hydrogen ceases. After cooling, the water and tincture are added. A "shake" label should be used, since a dark precipitate will slowly form.

**Saline Solution for Injections, Extemporaneous Preparation of.** Toege. (*Muench. Med. Woch.*, 1914, 1325; *J. Pharm. Chim.*, 1914, 11, 32.) Perfectly sterile sodium chloride solution may be made from ordinary, or even contaminated water, by adding to it the calculated quantity of HCl and then neutralizing this boiling solution with NaOH with phenophalein indicator.

The free HCl absolutely kills all germs. This method is specially useful for preparing salvarsan injection. One hundred c.c. of HCl solution, 2 : 100, is heated to boiling, and when cooled to about blood heat 0.30 Gm. of salvarsan is added. Dilute NaOH is then added until the precipitate at first formed is redissolved.

**Sodium Salicylate Solutions, Discoloration of, by Alkalies.** H. G. Greenish and A. E. Beesley. (*Pharm. J.*, 1915, **40**, 201.) From experiments made with pure sodium salicylate, prepared both from the synthetic and natural acid, it is shown that the appearance of the dark colour observed in alkaline aqueous solutions is not due to any impurity in the salicylate. In the case of solutions containing sodium bicarbonate and salicylate the cause is attributed to the action of oxygen in the presence of sodium sesquicarbonate,  $\text{Na}_2\text{CO}_3\cdot\text{NaHCO}_3$ . The last named salt is found to produce the dark colour and precipitate very rapidly with sodium salicylate solutions. The addition of a reducing agent retards the change. The addition of a grain of  $\text{Na}_2\text{SO}_3$  or  $\text{NaHSO}_3$  to an ordinary 8 oz. mixture has a marked effect in preventing the formation of the colour.

**Sodium Salicylate Solution, Discoloration of.** W. Macadie. (*Pharm. J.*, 1915 [4], **40**, 355.) The author attributes the darkening and precipitation of salicylate solutions to the presence of a trace of  $\text{NH}_3$  in the air. In large standards of stock solution of sodium salicylate, discoloration may be prevented by floating on the surface a small dish containing cotton wool saturated with acetic acid.

**Sodium Salicylate Solution, Discoloration of.** R. Welford. (*Pharm. J.*, 1915 [4], **40**, 512.) This may be obviated by the addition of  $\frac{1}{8}$  grain of salicylic acid to each fluid ounce of solution, and storing in amber glass bottles. It is stated that with this addition, the solution does not darken on adding alkali carbonates. Usually, this small amount of free salicylic acid would be quite unobjectionable.

**Strontium and Bismuth Mixture, Incompatible.** W. Sharmán and J. Smith. (*Pharm. J.*, 1914 [4], **39**, 443, 485.) The following was found impossible to dispense so as to form a presentable mixture : Strontii brom.,  $\mathfrak{z}\text{ii}$ .; liq. bism. am. cit.,  $\mathfrak{z}\text{vi}$ .; sp. chlorof.,  $\mathfrak{z}\text{iss}$ .; tr. calumb.,  $\mathfrak{z}\text{iss}$ .; aq., ad  $\mathfrak{z}\text{vi}$ . This is a case of chemical incompatibility. The ammonio-citrate

of bismuth is decomposed and the citrates of Bi and Sr are precipitated. It cannot be dispensed as written without both the Bi and Sr being precipitated.

**Sulphur Lotion.** (*Nat. Drugg.*, 1915, 49, 68.) The following prescription may be difficult to dispense with the sulphur and camphor evenly distributed: R Sulphur precip.,  $\mathfrak{Zi}$ .; camphor, gr. xv.; tragacanth, gr. xxv.; liquor calcis,  $\mathfrak{Zij}$ .; aquae dest., ad  $\mathfrak{Ziv}$ . M. Place the camphor in a mortar and powder it with the aid of a few drops of alcohol; then add about  $\mathfrak{Zij}$  more alcohol to dissolve it. Add the tragacanth, in fine powder, and mix intimately. Then add water,  $\mathfrak{Zij}$ ., in four portions with continual trituration. The sulphur is added to the gelatinous mixture,\* with further continuous trituration. The lime water is slowly added, and, finally, sufficient water to produce the prescribed volume. Precipitated or washed sulphur should not be substituted for the sublimed sulphur prescribed.

**Thymol Iodide Ointment.** (*Midland Drugg.*, 1915, 49, 195.) Ointment of thymol iodide seems to cause trouble to many dispensers on account of the difficulty in making a smooth ointment by simple trituration. The following method will ensure a smooth, homogeneous ointment. Place the thymol iodide in a perfectly dry mortar, add for each grain about 1 minim of ether, or sufficient to dissolve it. Then add about  $\frac{1}{2}$  minim of liquid paraffin for each grain and triturate to facilitate the spontaneous evaporation of the ether which usually takes place in about 5 minutes. Then the diluent, whatever it may be, is added and stirred thoroughly. Another method is to add a small amount of the diluent directly to the thymol iodide-ether mixture, prepared as directed, and allow the ether to evaporate as above.

**Thymol Iodide Ointment.** — Loustallot. (*Bull. Soc. pharm. de Bordeaux; Répertoire*, 1915, 27, 1.) Di-iodothymol does not mix well when rubbed down with vaseline, since it aggregates and clots. It should be first worked to a cream with a little liquid paraffin and then incorporated with vaseline.

**Thymol Iodide Ointment.** A. Boileau. (*Bull. Soc. Pharm. de Bordeaux; L'Union pharm.*, 1915, 56, 17.) When preparing aristol in ointment form, the best method is to dissolve it in just

sufficient  $\text{Et}_2\text{O}$  and to add this solution to the fatty base, and then mixing quickly until all odour of  $\text{Et}_2\text{O}$  has disappeared. The following is a typical ointment which may be dispensed thus : Aristol, 3 Gm. ; vaseline, 6 Gm. ; lanoline, 6 Gm. Six to 7 c.c. of  $\text{Et}_2\text{O}$  are sufficient to dissolve this amount of aristol.

**Zinc Sulphate and Borax in an Eye Lotion.** (*Chem. & Drugg.*, 1914, 85, 73.) Sod. bibor., gr. xx. ; zinci sulph., gr. vj. ; aq. rosae dest., ad  $\mathfrak{z}$ vj.—M. Sig. : "The Eye-lotion." To be used with an eye-cup. A precipitate of borate of zinc is produced in this eye-lotion. If the triple rose-water is diluted with a "hard" tap-water,  $\text{CaSO}_4$  and  $\text{ZnCO}_3$  would also be precipitated on standing, depending on the hardness of the water. If the two salts are dissolved separately in 3 fluid ounces of the rosewater and the solutions mixed, a finely divided precipitate will be obtained, and the lotion may be used safely. In some pharmacies, however, it is the rule to send out all eye-lotions perfectly clear, in which case the above would require to be filtered, but the procedure cannot be justified without reference to the prescriber, who might be told that the clear lotion is a solution of sodium sulphate and borate and zinc borate, the last in very small amount.

## GALENICAL PHARMACY

**Agar-agar, Flake, Preparation of.** F. W. Nitardy. (*J. Amer. Pharm. Assoc.*, 1915, 4, 174.) Agar-agar has recently come into favour with the medical profession as an evacuant. Its value is based on the formation of a soft, bulky indigestible jelly in the intestines, which, it is claimed, promotes peristalsis by supplying the necessary residue frequently lacking on account of our present-day highly refined foods, and softens the faeces by virtue of its water absorbing and holding power. In its natural form agar-agar is hardly available for this purpose as the long shreds are difficult to administer. The powdered agar-agar, it is claimed, will be digested when taken. Its most desirable form for administration is therefore in flakes, in appearance quite similar to flake breakfast foods and usually prescribed to be eaten with or as such cereals. A simple and easy method of preparation is as follows : Soak and rinse a suitable quantity of agar-agar in water, drain well, grind through a meat-chopper and spread out in thin layers on cheese-cloth trays to dry in a

dust-free airy place. When dry collect and store in suitable vessels. This product is usually prescribed in doses of one to four heaping teaspoonfuls. If it is desired to medicate the agar-agar, the required amount of medicament for each 500 Gm. is dissolved in water so as to form 1,000 c.c. of solution. This solution is mixed with the flake agar-agar, and as soon as it is evenly and completely absorbed, the product is again spread out to dry.

**Antiformin Substitute.** W. J. Matthews. (*Lancet*, 1915, 185, 356.) A perfect substitute for "antiformin" may be obtained by passing Cl into 15 per cent. solution of NaOH nearly to saturation. About 1 litre may be made thus at a cost of thirteen pence. This quantity will require 1 lb. of HCl sp.g. 1.160 and about 6 oz. of  $MnO_2$  to generate the requisite Cl.

**Antiseptic Dressing for Embalming Wounds.** L. Mencièrre. (*Lancet*, 1915, 188, 269.) The following solution, "A," is recommended for soaking strips of gauze for the permanent dressing of septic wounds: Iodoform, 10 Gm.; guaiacol, 10 Gm.; eucalyptol, 10 Gm.; Peruvian balsam, 30 Gm.; ether, 100 Gm. This dressing is to be renewed once daily. A weaker solution, "B," with the same ingredients but with ten times as much ether, is used to wash out the wound. This treatment gives excellent results with infected wounds and rapidly promotes healthy granulation.

**Bismuth Glycerite, Improved Formula for.** W. L. Scoville. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1294.) Bismuth subnitrate, 156 Gm.; nitric acid, 148 c.c.; tartaric acid, 232 Gm.; sodium bicarbonate, 325 Gm.; glycerin, distilled water, each a sufficient quantity to make about 1,000 c.c. Mix the  $HNO_3$  with 300 c.c. of distilled water, in a bottle having a capacity of about 4,000 c.c., and dissolve the  $BiONO_3$  in the mixture. Then slowly add 600 c.c. of distilled water and dissolve 116 Gm. of  $H_2C_4H_4O_6$  in the mixture. Now add, in small portions, 195 Gm. of  $NaHCO_3$ , shaking frequently and avoiding loss by effervescence. When all is added fill the bottle with distilled water and mix well. Allow the magma to settle and decant the clear liquid. Wash the magma by decantation, until the wash-liquor has but a slight saline taste. Then pour upon a filter and allow to drain, rinsing the bottle with a little water. Now transfer the moist magma to a porcelain evaporating dish and add 116 Gm. of  $H_2C_4H_4O_6$ , then slowly, and in small portions,

130 Gm. of  $\text{NaHCO}_3$ . Heat the mixture on a steam-bath until solution is effected and the total volume is reduced to 475 c.c. Then add 475 c.c. of glycerin, if necessary, enough water to make 950 c.c. of solution, and filter. Estimate the amount of Bi in the liquid by the process given below, and adjust it by evaporation or the addition of equal values of glycerin and water to contain the equivalent of 12.8 Gm. of  $\text{Bi}_2\text{O}_3$  in each 100 c.c. A colourless or pale yellow liquid having a slight odour of glycerin and a sweet, followed by a saline taste. Sp g. about 1.378 at  $25^\circ\text{C}$ . If 5 c.c. of the glycerite, accurately measured, be diluted to about 200 c.c. with water, this solution saturated with  $\text{H}_2\text{S}$  and allowed to stand 2 hours, then the precipitate collected on a tared filter, washed thoroughly with  $\text{H}_2\text{S}$  solution, then with a little EtOH, and finally with recently distilled  $\text{CS}_2$  until all free S is removed, then dried to constant weight at  $100^\circ\text{C}$ ., the residue should weigh not less than 0.690 Gm. The weight obtained multiplied by 0.905 and th's product by 20 gives the equivalent of  $\text{Bi}_2\text{O}_3$  per 100 c.c.

**Bismuth Preparations, B.P.C.** F. Goldby. (*Pharm. J.*, 1915 [4], 40, 826.) *Mist. Bismuthi Co. and Mist. Bismuthi Co. c. Morphina*. In these, an equivalent proportion of carmine should be used in place of the liq. carmini, B.P.C.; this can be readily dissolved in a portion of the ammonia solution. Since the publication of the Codex in 1911 a soluble bismuth tartrate in scales has been available. This may be used with advantage in place of the bismuth citrate and ammonia in the preparation of the *Mist. Bismuthi Co. c. Pepsino*. The following formula is suggested: Bismuth tartrate, soluble scales, 800 grains; stronger glycerin of pepsin, B.P.C.,  $2\frac{1}{2}$  fl. oz.; solution of strychnine, B.P.,  $1\frac{1}{2}$  fl. oz.; tincture of cudbear, B.P.C.,  $1\frac{1}{2}$  fl. oz.; dilute hydrocyanic acid, 320 minims; chloroform water (1 in 200), to 20 fl. oz. Dissolve the bismuth tartrate in  $12\frac{1}{2}$  fl. oz. of the chloroform water; add the remaining ingredients, and make up to 20 fl. oz. with chloroform water. The bismuth tartrate dissolves completely in about 1 hour, with frequent shaking, and the finished mixture is perfectly bright, needing no filtration, and is of a permanent character. The reaction is slightly acid, consequently the pepsin is more likely to be in active condition than when in alkaline solution, and on account of this acidity cudbear appears to be a more suitable colouring agent than carmine.

**Blaud's Pills, Commercial, Quality of.** L. E. Warren. *J. Amer. Pharm. Assoc.*, 1915, 4, 624.) The examination of fifteen commercial specimens of Blaud's pills, prepared by leading wholesale American firms shows that uniformity of dosage is not satisfactory. The amount of ferrous iron present calculated as  $\text{FeCO}_3$  ranged from 77 to 182 per cent. of the amount claimed. Only four of the samples contained less ferrous iron than was equivalent to the prescribed amount of  $\text{FeCO}_3$ . The results of the examination refute the commonly assumed instability of ready-made Blaud's pills. On the other hand, it is seen that the Blaud's pills of the market are not very reliable as to iron content. A range of from 77 to 182 per cent. of the claimed amount of ferrous carbonate denotes carelessness in manufacturing or lack of proper analytical control over the finished product. Further, the examination demonstrates that the "nascent" preparations, the soft mass pills, and the gelatin encapsulated oily suspension show no advantage over the ordinary kinds. In view of the findings, physicians should direct the pharmacist to prepare Blaud's pills according to the U.S.P. whenever they are prescribed.

**Calendula Cerate.** (*Nat. Drugg.*, 1915, 45, 124.) Lard, fresh, 8 oz.; fluid extract of calendula, 1 fl. oz. Heat on a water-bath until the EtOH has evaporated, stirring frequently meanwhile. Another method consists in digesting the flowers with melted lard for about 10 minutes, stirring occasionally, then strain and stir frequently until cooled. It is advisable to add about 2 ounces of yellow wax.

**Camphor and its Preparations.** J. L. Lascoff. (*Drugg. Circ.*, 1914, 58, 613.) After dealing with the history of the drug, the various official and unofficial preparations of camphor are enumerated, some of which are discussed in detail. The following camphor-containing formulæ are selected from those given. *Collyrium astringens luteum Violii*.—Ammonium chloride, 1.0; zinc sulphate, 2.5; dissolve in distilled water, 400.0; add camphor, 0.8; dissolved in diluted alcohol, 40.0; then add saffron, 0.2. Macerate 24 hours and then filter. *Aqua cosmetica Kummerfeldi*.—Formula No. 1: Sublimed sulphur, 1 part; spirit of camphor, 2 parts; spirit of lavender, glycerin, of each, 5 parts; cologne water, 10 parts; distilled water, 60 parts. A small quantity of acacia may be used to suspend the camphor in fine subdivision. Formula No. 2: Precipitated

sulphur, 10·0 Gm. ; camphor, 1·0 Gm. ; acacia, 2·0 Gm. ; lime water, rose water, of each, 150·0 Gm. *Emulsio camphorata*.—Powdered camphor, 1·0 Gm. ; acacia, 5·0 Gm. ; diluted alcohol, q.s. ; powdered sugar, 25·0 Gm. ; emulsion of almond, 250·0 Gm. *Unguentum hæmorrhoidale*.—Powdered saffron, powdered camphor, of each, 1·0 Gm. ; infused oil of hyoscyamus, 7·5 Gm. ; lead ointment, 20·0 Gm.

**Cherry Laurel Water, Preparation of.** M. Bridel and N. Delabrière. (*J. Pharm. Chim.*, 1915, 11, 110.) The authors do not agree with de Saint-Serin, who states that cherry laurel water may be best distilled from whole leaves. They find that a markedly larger product, richer in HCN, is obtained if the leaves are first chopped up. Even on the industrial scale, the yield is so far greater that it pays for the time and labour expended in disintegrating the leaves. In any case, if whole leaves are used, it is necessary to subject them to a process of maceration, previous to distillation. (See also *Y B.*, 1912, 305, 306 ; 1914, 188, 235.)

**Chlorinated Lime Tablets for sterilizing Drinking Water.** M. Vincent and M. Gaillard. (*J. Pharm. Chim.*, 1915, 11, 271.) The authors advocate the use of chlorinated lime as a most convenient and effective means of sterilizing drinking water, especially serviceable for troops in the field. As a soluble excipient sodium chloride is recommended as being the best salt. The quantities used to prepare a tablet to sterilize a litre of water are : chlorinated lime, 0·015 Gm. ; sodium chloride, 0·08 Gm. Each tablet when freshly made contains 0·0035 Gm. of available chlorine. In time, this falls to 0·003 Gm. But this small loss is very slow. In four months it amounted to only 0·0004 Gm. The addition of the salt has a very markedly favourable influence on the diffusion and solution of the chlorine. On adding one of these tablets to a litre of water and shaking, three-fourths of the available chlorine is liberated in 10 minutes, although the form of the tablet does not appear to be modified. In another 10 minutes almost the whole of the remaining chlorine is liberated. There is no need to crush the tablet. It may retain its form for several hours, due to the formation of a skeleton of calcium carbonate. This is not a disadvantage, in practice ; it enables the fact that any one in a series of rations of drinking water has been properly treated to be recognized at a glance. If the tablets are made



without sodium chloride they much more slowly give up their active chlorine. Besides the above quantities for 1 litre, tablets are made for sterilizing 5 and 10 litres of water; these containing respectively 0.075 and 0.150 Gm. of chlorinated lime, and 0.380 and 0.60 Gm. of sodium chloride. The convenience of this method for purifying water is evident. These tablets do not attack well tinned water bottles. If the tinning is defective, traces of ferric hydrate will be formed in suspension, which is of no importance from a hygienic point of view. Although experiments show that the tablets keep well for several months, as a precaution a fresh supply should be issued every two months.

**Cod Liver Oil in Emulsions, Determination of.** G. Bue m - m i n g. (*Apoth. Zeit.*, 1914, **29**, 695-7; *Chem. Abstr.*, 1915, **9**, 121.) Into a 50 c.c. Erlenmeyer flask weigh 1-2 Gm. of emulsion, add 10 c.c. 12.5 per cent. HCl and a small piece of pumice, heat gently over a wire gauze until a clear solution results, partially closing the flask the while with a small funnel. Pour the warm liquid into a 100 c.c. cylinder (graduated in 0.5 c.c. and having an inner diameter of 20 mm.), wash out the flask on cooling with 30 c.c. each of Et<sub>2</sub>O and petroleum ether, adding these solvents to and shaking vigorously with the treated emulsion. After standing 1-2 hours, note volume of ethereal solution, then transfer with a pipette 50 c.c. of the latter to a tared 150 c.c. round-bottomed flask, the pipette being subsequently washed out with a small quantity of petroleum ether in order to recover all traces of fat contained therein. Dissipate the solvent by distilling to dryness at 100°C. and weigh.

**Cod Liver Oil Emulsion, Determination of Oil in.** S c r i b a. (*Apoth. Zeit.*; *Drugg. Circ.*, 1914, **58**, 667.) Ten Gm. of the diluted emulsion (1 in 10) is mixed with 2 c.c. of AmOH, 10 c.c. of absolute EtOH, 20 c.c. of Et<sub>2</sub>O and 20 c.c. of petroleum ether, shaking the mixture well after the addition of each ingredient. The mixture is allowed to stand for 15 minutes, the aqueous liquid is drawn off and the ethereal layer is shaken with 0.6 Gm. of tragacanth. After clearing, the ethereal liquid is filtered into a tared beaker, the tragacanth washed with two portions of each 5 c.c. of petroleum ether, which is also filtered into the beaker. The combined ethereal liquids are evaporated and the residue is dried at 100°C. to constant weight.

**Cod Liver Oil Emulsion, Value of various Flavourings for.**

G. M. Beringer, Jr. (*Am. J. Pharm.*, 1915, **87**, 115.)

(1) Oils of coriander, geranium, anise and cardamom completely cover the fishy flavour of the cod-liver oil so that it is not apparent to taste or smell. (2) Oils of bitter almond, cloves, pimento, and vanillin only partly cover the taste and odour of the cod-liver oil but are very persistent and might prove of value blended with some of the flavours in Group 1. (3) Oils of peppermint, spearmint, lemon, orange and Ceylon cinnamon stand out very strongly and almost completely mask the odour, but not the fishy taste. (4) Oils of caraway, cassia, sassafras, wintergreen, betula, and nutmeg seem actually to accentuate the fishy taste. This is particularly true of nutmeg and wintergreen which comes out strongly both in taste and odour but has no effect whatever in covering the fishy taste. Coumarin is of no value as a blend.

**Cotton, Absorbent, Characters and Tests for.** A. L a h a c h e. (*L'Union pharm.*, 1915, **56**, 98.) Good absorbent cotton should be absolutely white, without any yellowish or pinkish shade; odourless, and tasteless. The texture should be even and the strands uniform and free from lumps or knots. It should not crackle when pressed between the fingers, and should not be friable and must contain no dust or small particles. These may be detected by shaking a sheet of the cotton in a dark room into which a beam of light penetrates. When pulled out so as to obtain straight threads, these should measure not less than 3 Cm. The measuring is conveniently performed against a black surface. The fibres should have a fair tenacity; when a pinch is taken between the thumb and index finger of each hand, a distinct force should be necessary to break the cotton. Inferior cotton is easily broken thus. A tuft of cotton floated on the surface of water should sink instantly to the bottom of the container. Inferior qualities will float for varying periods. Any cotton which takes more than 3 seconds to sink to the bottom of the vessel should be rejected. The absorptive power may be determined thus: A sheet of thoroughly dried cotton is cut in a square to weigh 10 Gm. This is put in a tared capsule of 1 litre capacity, and moistened with a litre of water. After 10 minutes' contact, the excess of liquid is decanted, and the cotton drained without pressure or moving for exactly 5 minutes. The increase of weight is then determined. The amount, divided by 10, gives the coefficient of absorption for

the cotton. This should not be below 18. When a flake of cotton is burnt, the whole surface should at once be consumed without any carbonaceous residue. Cotton of good quality should not contain more than 5 per cent. of moisture. When 10 Gm. of cotton is macerated for an hour in 100 c.c. of warm water, and expressed; the liquid should be quite neutral to litmus; should leave no appreciable residue on evaporation, and what little residue is left should not blacken when heated. Macerations with  $\text{Et}_2\text{O}$  and with  $\text{EtOH}$  should give similar results.

*Absorbent gauze* should have similar characters to absorbent cotton. The determination of the amount of water absorbed is, however, less definite on account of the variability of the size of the meshes. The most generally useful mesh is  $15 \times 15$  threads in the  $\text{cm}^2$ . (See also *Y.B.*, 1906, 115; 1912, 308.)

**Cottons and Dressings, Medicated, Surgical, Simple Tests for.**—*Lahache*. (*L'Union pharm.*, 1915, 56, 145.) *Sublimate Cotton and Dressings*.—A particle of the material in a saucer is moistened with dilute  $\text{H}_2\text{S}$  solution. The fibres should be blackened immediately and uniformly without prominently darker patches or points. Another particle, similarly moistened with dilute  $\text{AmOH}$ , will show more or less darkening due to presence of calomel. The amount observed will indicate the degree of alteration which the material has undergone. When calcined, the dressing should burn without blackening; tartaric acid if present will be indicated by its characteristic odour on charring. Sublimate dressings should not be more than 6 months old and should be carefully stored, since  $\text{HgCl}_2$  is volatile at ordinary temperatures. *Iodoform Dressings*.—Water in which iodoform dressing has been macerated should show no evident colour. If it is tinted yellow, sophistication may be suspected. Dressing macerated in  $\text{Et}_2\text{O}$  or  $\text{CHCl}_3$  should be completely decolorized, the solvent being coloured. The intensity of the colour obtained with definite quantities of material will give a rough indication of the strength in iodoform. Iodoform dressings should not be more than 6 months old. *Salol Dressings*.—These should be quite white and have an odour recalling meadowsweet. If a piece of the dressing is first moistened in  $\text{EtOH}$  and then immersed in water containing 5 drops of  $\text{Fe}_2\text{Cl}_6$  the violet colour produced should be evenly distributed over the whole piece and

the intensity of colour will afford a rough indication of the amount of salol present. If a piece be first moistened with water, instead of with EtOH as above, and then plunged in the  $\text{Fe}_2\text{Cl}_6$  solution, it should not be coloured violet at once. If this occurs it indicates that salicylic acid has been fraudulently substituted for salol in the dressing. *Boric Acid Dressings*.—A piece soaked in EtOH and a few drops of  $\text{H}_2\text{SO}_4$  should give a green flame when ignited. A piece of the fabric is spread out on a flat surface and a sheet of paper moistened with a dilution of tincture of turmeric, 1 : 10, and acidified with a few drops of HCl is lightly pressed over the surface and removed. On drying the points where the paper has touched the fabric should show evenly distributed red-brown spots. *Salicylic Acid Dressing*.—When immersed in very dilute aqueous solution of  $\text{Fe}_2\text{Cl}_6$  pieces of the dressing should assume an even violet colour. *Zinc Peroxide Dressing*.—A piece of the fabric immersed in a 1 : 1,000 solution of  $\text{KMnO}_4$  with a few drops of  $\text{H}_2\text{SO}_4$  should immediately decolorize the liquid. The uniformity of distribution may be tested by moistening a piece of the fabric with a mixture of KI solution 1 : 10 and  $\text{H}_2\text{C}_2\text{O}_4$  1 : 10 in equal volumes. The blue tint produced should be uniform, and the intensity gives a rough indication of the strength. *Bismuth Subgallate Dressing*.—The liquid in which a piece of the fabric has been macerated, either water, EtOH or  $\text{Et}_2\text{O}$ , should not blacken when treated with  $\text{H}_2\text{S}$  solution. HCl should instantly decolorize the dressing and the acid liquid should give a black precipitate with  $\text{H}_2\text{S}$  and also with  $\text{Fe}_2\text{Cl}_6$  at  $100^\circ\text{C}$ . The above tests are intended for extemporaneous use only and to be supplemented by chemical determinations.

**Cotton Wool, How it is Made.** (*Chem. & Drugg.*, 1914, 85, 452.) An illustrated article describing the machinery and processes used in the manufacture of cotton wool.

**Creams, Medicated, for Administering Drugs in the Field.** F. W. Tunncliffe. (*Lancet*, 1915, 188, 274.) The use of medicated creams, put up in collapsible tubes with standard nozzles, is suggested for the administration of necessary medicines to sick and wounded troops in the trenches or under cover. The dosage of these is so adjusted that the prescribed quantity of active ingredient is contained in 1 linear inch of the cream discharged on pressing the tube. For instance, creams containing 30 minims of chlorodyne or 2 grains of morphine hydro-

chloride in the linear inch have been prepared. The consistence of any desired basis may be modified by the use of glycerin, agar-agar or bismuth paste to meet climatic variation. An aromatic should be prescribed as well to stimulate salivary secretion and thereby assist swallowing the dose.

**Digitalis, Alcoholic Strength of Galenical Preparations of.** R. A. Hatcher. (*Drugg. Circ.*, 1914, 58.) Tinctures and fluid extracts should not only be made with alcohol 70 per cent., but the finished products should actually contain that amount of alcohol. If this is assured, the preparations will undergo no important deterioration of potency on keeping almost indefinitely.

**Distilled Aromatic Waters and the Composition of the Essential Oils therein.** A. Goris and C. Vischniac. (*Bull. Sci. pharm.*, 1915, 22, 66.) It is generally assumed that because aromatic distilled waters are saturated solutions of the essential oils of the drug from which they are distilled, that the chemical composition of this water-soluble portion is identical with that of the essential oil. The authors show that this is far from being the case. Most of the essential oils are complex mixtures: some of the constituents are much more soluble than others. In the case of *cinnamon water*, after filtering the milky aqueous portion through moistened paper to remove the suspended oil, saturating this clear filtrate with NaCl, and shaking out with Et<sub>2</sub>O, the latter on evaporation left an essential oil yielding 92 per cent. of aldehyde by the bisulphite method. The oil deposited on the bottom of the receiver during the distillation of this cinnamon water was found to contain only 75 per cent. of aldehyde. With *thyme* a similar difference occurred between the water-soluble oil and that which separated and floated on the surface during the process of distillation. The water-soluble oil gave 46 per cent. of phenols, mainly carvacrol. The insoluble oil contained 36 per cent. of the same phenol.

**Elixir Acetomorphinae et Terpini B.P.C.** (*Pharm. J.*, 1915 [4], 40, 686.) On standing, the sugar in the syrup of wild cherry crystallizes out, on account of the alcoholic strength of the preparation. Liquid extract of wild cherry and gluside elixir should be substituted.

**Emulsion of Oil of Cade.** A. Otto. (*Nat. Drugg.*, 1914, 44, 294.) The best emulsion of cade oil, to use as an addition to water for washing and bathing, is prepared according to the

following formula : Oil of cade, 50 Gm. ; extract of quillaia, 5 Gm. ; yolk of one egg, No. 1 ; water, 250 c.c.

**Emulsions, Determination of Oil in.** C. H. La Wall and L. Forman. (*J. Am. Pharm. Assoc.*, 1914, 3, 1444.) The following is a modification of the method of Gottlieb and Rose. Prepare a mixture of the emulsion in distilled water, so that each 100 c.c. of the liquid contains 40 Gm. of the emulsion. Take two 100 c.c. graduated cylinders and in one place 10 c.c. of the diluted emulsion and in the other place 5 c.c. of the diluted emulsion and 5 c.c. of water. To each cylinder then add the following reagents in the order named, agitating thoroughly after each addition : 1 c.c. of AmOH, sp.g. 0.897 ; 10 c.c. of EtOH 95 per cent. ; 25 c.c. of Et<sub>2</sub>O, sp.g. 0.717 ; 25 c.c. petroleum benzin, b.p. 45°–60°C. After the addition of the petroleum benzin, the agitation should be continuous for 10 minutes, after which the cylinders should be allowed to stand until the liquids have separated into two layers with a sharp dividing line (this requires from 15 minutes to 1 hour). Then having observed the exact volume of the upper layer, draw off exactly one-half and transfer to a flat-bottomed glass capsule and evaporate quickly on a water-bath to constant weight. In one of the duplicates the resulting fat will be from 2 Gm. of the emulsion, in the other from 1 Gm., which gives a satisfactory check upon the thoroughness of the extraction.

**Fatty Gauze for Dressing Wounds.** Lumière. (*Répertoire de Pharm.*, 1915, 27, 162.) The inconvenience and discomfort caused by dressings sticking to the surface of the wound may be avoided by the following simple device. Gauze with 2 mm. meshes is impregnated with vaseline, or with a mixture of beeswax, vaseline, castor oil, and Peruvian balsam. This is laid on the surface of the wound, then absorbent cotton or other dressing is superimposed. All the wound secretions have a free passage through the gauze, and the whole dressing is easily taken off without disturbing the granulating tissue.

**Ferrated Cod Liver Oil, Preparation of.** C. E. Carlson. (*Svensk. Farm. Tid.* ; *Chem. Abstr.*, 1915, 9, 352.) The fatty acids of oil of sweet almonds readily combine with Fe, forming a clear stable red-brown liquid which contains 5.6 per cent. of Fe. This is odourless and tasteless, and does not congeal in cold weather. It is readily soluble in cod liver oil.

Ferrated cod liver oil is prepared by mixing 1 part of this iron solution with 9 parts of cod liver oil.

**Fluid Extracts, Amount of Extractive in.** E. L. Maines and R. J. Gardner. (*J. Amer. Pharm. Assoc.*, 1914, 3, 997.) The authors insist on the importance of the determination of the total extractive in the control of preparation of fluid extracts, especially of non-alkaloidal drugs. The average percentage yields of extractive in over 170 fluid extracts from different drugs are given.

**Fluid Extracts, Manufacture of.** C. F. Ramsay. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1646.) On the large scale difficulties are often met with in preparing fluid extracts which are not met with in small batches. Mucilaginous drugs give trouble in this respect. Admixture with sawdust or shavings is recommended to aid percolation. Fluid extract of squill is best prepared by extracting the cut, not powdered, bulb with alcohol 80 per cent. *Cereus grandiflorus* is also troublesome. The best method is to wash the drug with alcohol 95 per cent., concentrating these washings by distillation, and then extract the residual drug by percolation with more alcohol. Bladderwrack, calumba, lappa, and squaw vine are also difficult drugs to percolate. A strongly alcoholic menstruum should be used. Arnica flowers, colocynth, quassia, red clover, sourwood leaves and marrubium require to be packed very tightly in the percolator to ensure proper extraction. Hard drugs such as physostigma, podophyllum and stone root require to be in very fine powder. The addition of glycerin to the menstruum often retards percolation. Trouble is thus occasioned with uva ursi, geranium, hydrastis, rose, and cinchona. Some drugs very rich in extractive give trouble at first from the very thick percolate. Such are poplar buds, black willow bark, elder flowers, helonias, digitalis, and mistletoe. With these it is best to employ a number of small percolators for a batch and to reserve a portion of the first percolate from each. Drugs rich in oil, such as East Indian sandalwood and sabal, will give a cloudy first percolate, due to dilution of the alcohol, 95 per cent., by the soluble matter in the drug; this throws out the oil from solution. This portion should be concentrated in the still, and the percolation continued with alcohol 95 per cent. Convallaria and digitalis cannot be properly extracted by the alcoholic menstruum of the U.S.P. Alcohol 80 per cent. gives better results. With alka-

loidal drugs, prolonged percolation is often necessary to obtain complete exhaustion. In the case of calisaya bark, better results are obtained by percolating at first with alcohol alone and not adding glycerin to the menstruum. Sanguinaria is best extracted with alcohol 71 per cent., containing 2 per cent. of HCl, the drug being coarsely powdered. Gelsemium, nuxvomica, pilocarpus, sundew, echinacea, fringe tree, gravel plant, adonis, arbor-vitae, and caulophyllum are enumerated as being difficult to exhaust. Each drug requires individual treatment, and often a special menstruum, in order to obtain a fully active elegant fluid extract.

**Glycerin and Acetic Acid as a Substitute for Alcohol.** W. Wolstenholme. (*Pharm. J.*, 1915 [4], 40, 885.) The use of a menstruum composed of glycerin, 10; acetic acid (33 per cent.), 1; water, to 40, is recommended as a substitute for alcohol in the preparation of galenicals, such as tinctures. The name "glyncture" is suggested for these. The following are thus described: Glyncture Chiretta.—An excellent preparation. Glyncture Calumbæ.—Compares well with the tincture. Glyncture Hamamelis.—Yields a very slight deposit. Some made in 1903 was good in 1910. Glyncture Quillaia.—A failure, depositing rather badly. Glyncture Rhei Co.—A partial success, but needed fuller experiment. Glyncture Croci.—Satisfactory. Glyncture Quassia.—Very satisfactory. Glyncture Aurantii.—Not satisfactory. Deposits and aroma not good. Glyncture Capsici.—Not quite so good as B.P. tincture, but would probably work with a larger proportion of acid. An objection to these preparations is that they are slightly acid, and on exposure to sunlight after a day or two liberate iodine from potassium iodide. This should be borne in mind and provided against when using them with iodides and bromides. Another type of preparations made from extracts are worth attention.

**Glycerin in Galenicals, The Fungicidal and Antifermentative Action of.** G. J. Keller. (*Nat. Drugg.*, 1915, 45, 190.) For years the author employed the addition of from 5 to 10 per cent. of glycerin in the preparation of *Syrupus hypophosphitum*, *Syrup. hypophosphitum co.* and *Syrup. ferri iod.* It is also useful in N.F. preparations in place of sugar saccharin solution or syrup. A *Glyceritum hypophosphitum compositus*, in which the sugar is replaced by 50 per cent. (volume) of glycerin, is an improvement. This compound embraces both



stability and palatability with full retention of its therapeutic value, and is better fitted as a tonic for patients of diabetic tendencies than the U.S.P. syrup. Glycerin has been used in *Essentia Pepsini*, *Elixir Gentianae Glycerinatum* and *Elixir Terpini Hydratis*. There is perhaps no preparation in the N.F. which has caused more trouble to the pharmacist than *Essentia Pepsini*. The formula is as follows : Pepsin gran., 22.5 Gm. ; rennin, 16.5 Gm. ; lactic acid, U.S.P., 2.0 c.c. ; oil of Tangerine orange, 1.0 c.c. ; alcohol, 60.0 c.c. ; glycerin, 200.0 c.c. ; Moselle wine, 365.0 c.c. ; talcum, 15.0 Gm. ; water, q.s., to make 1000.0 c.c. Mix pepsin and rennin with about 200 c.c. of water, add the lactic acid and glycerin. Set aside for about 1 week, then add wine, orange oil, which previously has been dissolved in the alcohol, followed by sufficient water to make up quantity. Incorporate the purified talcum and filter after standing 3 or 4 days. The trouble maker in this product has been the wine and the syrup. Domestic wines or imported sherry wines are absolutely unfit to be used on account of the large amount of tannin they contain. An imported Moselle wine, properly aged and clarified, should be used in order to obtain good results.

*Elixir Gentianae Glycerinatum*, modified, contains most all of the ingredients as ordered in the N.F., with the exception of acetic ether solution of saccharin and sugar. Imported Duff Gordon sherry is used in place of the white wine ordered. Glycerin 50 per cent. (volume).

In *Elixir Terpini Hydratis*, modified, the alcohol has been reduced to 35 per cent., the syrup which is ordered in the N.F. and which is thrown down as a crystalline deposit on account of the large amount of alcohol present is entirely eliminated and glycerin substituted therefor. *Liquor Saccharini* is discarded as useless and 1 c.c. of bitter almond oil added as a flavouring to each 1,000 c.c. of elixir.

**Hypodermic Solutions, Preparation of.** P. Vadam. (*Bull. Sci. pharmacology*, 1915, 22, 86.) A series of articles, dealing with sterilization of the materials and apparatus, the solvents used, the action of heat on the solutions, and the unification of formulæ for injections.

**Iodine Ointment, Changes in.** L. H. Fried. (*J. Amer. Pharm. Assoc.*, 1915, 4, 621.) The gradual iodine absorption by the benzoated lard in this ointment has

been watched for a long period. Although considerable at first, the absorption gradually decreases until an equilibrium appears to be reached when just over 70 per cent. of the iodine originally added remains in the free state. It is stated that some prescribers prefer that the ointment should not be freshly made, since the older preparation is considered to be as active therapeutically and less irritant. The U.S.P. prescribes 4 per cent. of I for the ointment, with KI and glycerin. The following percentages of iodine were determined: Immediately after making, 3.89; 1 hour after making, 3.51; 1 day after making, 3.48; 5 days after making, 3.06; 10 days after making, 2.84; 30 days after making, 2.81; 90 days after making, 2.8096; 8 months after making, 2.8095 per cent.

**Iodotannin Syrup, Condition of the Iodine in.** C. Debreuil. (*Bull. soc. pharmacol.*, 1914, 21, 409-11.) Iodine exists in iodotannin syrup in the form of HI. When iodotannin syrup is dialyzed HI is found in the dialysate. When an iodotannate is treated with NaCl, HCl is formed according to the reaction  $HI + NaCl = HCl + NaI$ , and may be distilled off *in vacuo*. When iodotannates in diluted solution are treated with  $ZnCO_3$ , all the I is found in the solution as  $ZnI_2$ . This last reaction may be used in determining the I in iodotannates; if the solution is made up to a definite volume and the  $ZnCO_3$  added, an aliquot can be filtered off and the I determined by titration with N/10  $AgNO_3$  solution.

**Kola Extract, De-resinified.** E. Dufilho. (*Bull. Soc. Pharm. Bordeaux*, 1914, 53, 475, 524.) In the process of making kola extract it is only a slight advantage after distilling off the alcohol to evaporate the aqueous residue *in vacuo*, as the extract so obtained is no richer in alkaloid and only slightly less coloured. As evaporation proceeds in either case, a resinoid impurity separates which adheres to the bottom or sides of the pan. This should be removed by decanting the thin extract into another vessel. As a rule 30 per cent. of this resinous extract is present in ordinary kola extract. It contains from 3 to 7 per cent. of caffeine and may be used in another batch of the preparation. Although the yield of extract is considerably lessened by the removal of this resinoid impurity, its richness in caffeine is increased. Also the final product is soluble in all proportions in simple syrup, and almost soluble in water. It is therefore well adapted for the preparation of saccharine

granules and other preparations. In order to completely exhaust kola nut with EtOH, 60 per cent., it is necessary to percolate with twelve times its weight. This is best done by maceropercolation; macerating for 12 hours, then percolating for another 12 hours, alternately, until all the drug is exhausted.

**Lipiodin Ointment.** (*Schweiz. Apoth. Zeit.*, 1915, 53, 151.) Lipiodin, 1; sesame oil, 2; lanolin, 7. Dissolve the lipiodin in the sesame oil at 35°–40°C., then incorporate it with the lanolin.

**Liquid Paraffin Emulsion.** E. Pritchard. (*Practitioner; Pharm. J.*, 1914 [4], 40, 530.) The following is the formula the author invariably prescribes: R Paraffini liquidi, B.P., 33·00; acidi benzoici, 0·05; glusidi, 0·05; olei cinnamomi, 0·10; decoctum chondri crispī, ad 100·00. This can only be made satisfactorily in large quantities at a time—one gallon, at least.

**Liquor Cresolis Compositus, Improved Formula for.** E. L. Maines. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1325.) In the manufacture of compound solution of cresol, it is not advisable to follow the exact directions of the U.S.P.; the following formula and method of procedure having been found to produce better results: Cresol, 50; linseed oil, 35; potassium hydroxide, 8; water, q.s. Place the KOH in a steam-jacketed kettle or tank, equipped with an agitator, and add sufficient water to dissolve the KOH. Turn steam on kettle, keep agitator running, and add the linseed oil. Stir until the soap becomes clearly soluble in distilled water, adding small quantities of water from time to time in order to complete the saponification. Add the cresol gradually, constantly stirring the mixture until a clear solution is produced. Finally add sufficient water to make the desired yield, if necessary. It is not necessary to use any specified amount of water in the manufacture of compound solution of cresol, as any excess may be quickly evaporated in the steam kettle before adding the cresol. Any excess of water can easily be determined by noting the consistence of the soap. This is an important point to be observed, as it is the key to the successful manufacture of this product.

**Liquor Cresol Saponatus.** H. Finne more. (*Pharm. J.*, 1914 [4], 39, 661.) The use of castor oil in this pre-

paration, for preparing the soap base, is adversely criticized. Both the U.S.P. and the B.P.C. formulæ employ linseed oil. Both give clear solutions on dilution with water. The amount of castor oil used is thrice that of the linseed oil in the B.P.C. preparation. There is an unnecessary increase in cost, unless a corresponding advantage can be shown. The preparation made with castor oil has the serious disadvantage of giving a much more turbid solution when diluted with tap water than one compounded with linseed oil.

**Liquor Magnesii Citratis.** J. L. Brown. (*Nat. Drugg.*, 1914, 44, 377.) The following formula and method of manipulation is stated to give a satisfactory product: Magnesium carbonate, U.S.P., 180 Gm.; citric acid, 396 Gm.; syrup, 720 c.c.; spirit of lemon, 10 c.c.; potassium bicarb., 12-2.5 Gm. tablets; water to make 12 bottles of solution. Place the magnesium carbonate in an aluminium vessel of about 4 litres capacity, which contains about 2 litres of water. Now add the citric acid. Let stand till effervescence ceases and complete solution results, then allow to boil for a few moments. Add the spirit of lemon and filter while hot. When the solution has all passed, wash the filter with 550 c.c. of boiling water through it. Now add the syrup and divide the liquid accurately between twelve patent-stoppered citrate bottles. Fill the bottles nearly full with water, drop in each a 2.5 Gm. tablet of potassium bicarbonate and stopper immediately.

**Mercuric Chloride Tablets and Legislation relating thereto.** G. M. Beringer. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1111). A review is first given of the official formulæ in various European pharmacopœias. A mixture containing equal parts of  $\text{HgCl}_2$  and  $\text{NaCl}$  is considered to be the most useful and stable form for the preparation of the tablets. The name "*Toxibellæ*" is suggested for these. Indigo carmine is recommended as the colouring agent; from 0.0025 to 0.005 Gm. may be used for each tablet. The official tablet should be adjusted to a strength of 0.5 Gm. of  $\text{HgCl}_2$  in each so that it will give with 500 c.c. of water a 1:1,000 solution. The shape of the tablet should be distinctive to prevent misadventure. The coffin shape, suggested by Alpers, is to be commended. The shape of the Ph.G. official tablet, twice as broad as long, and its red colouring are strongly condemned as closely resembling certain

popular laxative tablets. American legislation on the sale of these tablets is discussed.

**Nascent Iodine Dressing, Portable, for Campaigning.** Fonzes Diacon and A. Astruc. (*J. Pharm. Chim.*, 1915, 11, 123.) Pads of compressed absorbent cotton  $8 \times 10$  cm., say  $3 \times 4$  inches, are impregnated with KI and  $\text{KIO}_3$  solutions, dried and sterilized. These are wrapped in gauze, previously treated with a soluble acid. One of these pads is firmly sewn near the end of a surgical bandage, 3 metres long and 8 cm. wide (about 3 yards and 3 inches). The other pad is attached to the bandage by a single thread only, merely to prevent it falling to the ground when the bandage is unrolled. The whole is enclosed in a waterproof paper envelope, with a couple of safety pins. Two pads are provided, one for the entrance and the other for the exit wound. The fixed pad is applied to the former, and the loose one to the latter, the bandage applied in the usual way. If there is but one wound, both pads are applied to it. The blood issuing from the wound provides sufficient moisture to liberate the I from the  $\text{KIO}_3$ . This is kept in solution by the KI. There is no need to wet the dressing first.

**Pasta Iodi et Amyli, B.P.C.** P. B o a. (*Pharm. J.*, 1915 [4], 50, 485.) The original formula of Martindale for this preparation was as follows : Starch, 1 ; glycerin, 2 ; water, 6 ; solution of iodine, 1. Boil the first three together, and when nearly cold add the iodine solution. The B.P.C. 1907 is similar, but it directs loss by evaporation to be made up by adding water to make the final volume 10. The B.P.C. 1911 orders the first three ingredients to be heated on the water-bath until, on constant stirring, a paste is formed ; the solution of iodine is to be incorporated with the cool paste. As the starch grains are not properly disintegrated in this process, the product is even more liquid than that of the 1907 formula. The latter is not so firm as the product of the original formula, which is to be preferred.

**Pigmentum Chrysarobini.** G. B a r k e r. (*Pharm. J.*, 1914 [4], 39, 721.) The following is a satisfactory formula : Chrysarobin,  $\text{Ziii}$ .; chlorof. meth.,  $\text{Ziii}$ .  $\text{Zii}$ .; resin,  $\text{Zii}$ .; acid oleic,  $\text{Zi}$ .

**Pills and Granules made on the Manufacturing Scale.** M.

**François.** (*J. Pharm. Chim.*, 1915, 11, 275.) The author doubts if accurate and uniform dosage of pills and granules is attainable by purely mechanical means when large bulks of material are mixed and subdivided into minute quantities. It is not sufficient that exactly the amount of active ingredient should be weighed out for each batch. Analytical control of the finished preparations should be frequent. To facilitate this, it is important that the excipients employed should be simple, soluble and such as do not interfere with the determination of the active ingredients. All coatings, finishing, silvering, varnishing should be discouraged and the public should be educated to prefer their medicines in their natural state. For extremely active medicines the author would insist on the preparation of smaller quantities by hand of such articles as pills and granules.

**Pulvis Fluens Hydrargyri.** P. G. Unna. (*Dermatol. Woch.*, 1915, 337; *Chem. & Drugg*, 1915, 86, 744) When metallic Hg is rubbed down with oil of turpentine and lycopodium, the mercury is immediately "killed" and a dry mobile powder readily obtained containing 33 per cent. of Hg. The powder can be applied to the skin without the addition of fat, and in this form the mercury is an excellent parasiticide. It may also be employed as the means of preparing mercurial plasters and ointments. The author considers that its use will occasion a radical change in the external therapeutic use of Hg. Incidentally, the change occurring in old mercurial ointment was investigated. The specimen examined contained 0.9 per cent. of  $\text{Hg}_2\text{O}$ ; 1.76 per cent. of  $\text{HgO}$ ; 0.47 per cent. of mercurous oleate, and 3.83 per cent. of mercuric oleate. The known action of old mercurial ointment in "killing" metallic mercury is attributed to the minute globules being coated with a layer of oxide, which prevents their subsequent aggregation. Turpentine oil, acting as a carrier of oxygen, brings about this superficial and slight oxidation in a similar manner.

**Quiniolol, Iodized Powdered Cinchona Bark.** A. Mouchet and — Malbec. (*Paris médical; Répertoire de Pharm.*, 1915, 27, 83.) Iodine, 5 or 10 Gm., is dissolved in  $\text{Et}_2\text{O}$ , 100 or 200 Gm., and the solution triturated with finely powdered red cinchona bark, 100 Gm., until all the  $\text{Et}_2\text{O}$  is evaporated. The product, known as "quiniolol," is an effective antiseptic powder. The stronger preparation containing 10 per cent. of I is specially valuable as a dusting powder for gangrenous wounds. The

5 per cent. preparation is a useful iodoform substitute, being less costly, and devoid of unpleasant odour.

**Red Gum, Liquid Preparation of.** J. K. Thum. (*Amer. J. Pharm.*, 1915, 86, 449.) The following preparation keeps well and will replace any of the liquid preparations of red gum: Red gum, powdered, 200 Gm.; glycerin, 250 c.c.; water, a sufficient quantity to make 1,000 c.c. Mix the glycerin with 500 c.c. of water, and triturate the powdered red gum with sufficient of the mixture to produce a smooth paste. Transfer this to a flask by the aid of the remainder of the mixture of glycerin and water and heat on a water-bath for one hour; filter through purified cotton, keeping the funnel well covered. Finally, pass sufficient water through the filter to obtain 1,000 c.c. of fluid.

**Rum-Serum for Typhoid.** V. Courtellemont. (*Bull. Soc. Méd. de Paris; L'Union pharm.*, 1915, 56, 217.) The use of rum serum, similar to that suggested for post-operative collapse, is strongly recommended to counteract the adynamia of typhoid. The serum employed contains 47 Gm. of glucose per litre. It is sterilized, and the moment of use, 50 c.c. of rum is added to this quantity or 12.5 c.c. to each 250 c.c. flask. Old rum, of 40 to 48 per cent. of alcohol by volume [=30 to 16 under proof of the British Excise] is the spirit to be preferred for the purpose. The dose is 250 c.c. of rum serum daily or every other day; but twice this amount may be given without harm; as much as 1 litre in 24 hours has been used, administered in two separate injections.

**Sage, Galenical Preparations of.** P. E. Hommell. (*Nat. Drugg.*, 1915, 45, 164.) Sage is of undoubted therapeutic value. It has fallen into disuse except in domestic medicine, because no definite formulæ exist for its galenical preparations. The author supplies these:—

No. 1. *Fluid Extract of Salvia*.—Salvia in No. 60 powder, 1,000 Gm.; dilute alcohol, 49 per cent., to make 1,000 c.c. Proceed in usual manner.

No. 2. *Fluid Glycerate of Salvia*.—Salvia in No. 30 powder, 100 Gm. Proceed according to the type process (Beringer, *Y.B.*, 1909, 146), using 100 c.c. of the glycerin-water menstruum to moisten the drug. This is a satisfactory preparation of the drug, being clear and possessing a strong odour and taste of the herb, it mixes clear with water, syrup or dilute alcohol.

**No. 3. Tincture of Salvia.**—Salvia in No. 60 powder, 200 Gm. ; dilute alcohol, 49 per cent., q s., to make 1,000 c.c. Proceed in the usual manner

**No. 4. Syrup of Salvia.**—Sage, 50 Gm. ; glycerin, 100 c.c. ; sugar, 750 c.c. ; boiling water, 500 c.c. ; cold water to make 1,000 c.c. Upon the sage contained in a suitable vessel pour the boiling water and allow to macerate one hour ; then strain and add the glycerin, and pass as much cold water through the strainer until the liquid measures 500 c.c. Dissolve the sugar in the liquid by agitation.

**No. 5. Gargle of Salvia.**—Pulv. alum, 2 Gm. ; chlorate of potassium, 4 Gm. ; glycerin, 15 c.c. ; infusion salvia, q s., 250 c.c.

**No. 6. Ointment of Salvia.**—Solid extract of salvia, 10 Gm. ; hydrous wool fat, 50 Gm. ; white petrolatum, 40 Gm. Mix the extract of sage with the melted wool fat and petrolatum. Stir until cool. [The author does not give a method for preparing the "solid extract" of sage. Probably for incorporation in this ointment, the residue obtained by evaporation or distillation from the fluid extract would be satisfactory.—ED. Y.B.] Sage is popular in hair tonics and invigorators, and there can be no doubt but that in proper combination it increases the circulation of blood about the hair glands and roots so as to promote hair growth. It is effective when exhibited in the form of the tincture combined with resorcinol, glycerin and strong bay rum.

**Saline Antiseptic Solution.** C. H. La Wall. (*J. Amer. Pharm. Assoc.*, 1915, 4, 181.) The following is a "new type" of antiseptic solution containing no glycerin and practically no EtOH ; saline in taste and alkaline in reaction. It is an efficient mouth wash and nasal douche. Sodium chloride, 5 Gm. ; sodium borate, 5 Gm. ; sodium bicarbonate, 10 Gm. ; oil of spearmint, 1 c.c. ; oil of eucalyptus, 0.5 c.c. ; menthol, 0.1 Gm. ; alcohol, 5 c.c. ; fluid extract of hydrastis (aqueous), 2 c.c. ; water, q.s., to make 1,000 c.c. Dissolve the salts in 750 c.c., of water. Dissolve the oils and menthol in the EtOH. Mix the EtOH solution of the oils with 5 Gm. of MgCO<sub>3</sub> and triturate gradually with the aqueous solution of the salts. Filter and add the fluid extract of hydrastis, and finally add enough water through the filter to make 1,000 c.c.

**Salol with Liquid Paraffin.** F. Gold by. (*Pharm. J.*, 1915, [4], 40, 316.) As salol is perfectly soluble in liquid paraffin,



and the latter passes through the system practically unchanged, this would seem to be an ideal method of exhibiting salol as an intestinal antiseptic combined with the lubricating and laxative properties of the paraffin. The formula employed is as follows : Salol, 800 grains ; oil of cinnamon, 40 minims ; chloroform, 4 fl. drachms ; Biebrich scarlet,  $\frac{1}{2}$  grain ; liquid paraffin, to 80 fl. oz.

**Salvarsan, Glycerol-guaiacol Solution of.** G. Galvagni. (*Boll. chim. farm.*, 1914, **52**, 609 ; *Chem. Abstr.*, 1915, **9**, 123.) An improved vehicle for the hypodermic injection of salvarsan is prepared as follows : Pure guaiacol, 30 Gm. ; glycerin (twice distilled, diluted and sterilized), 2 Gm. ; and salvarsan, 30 Gm. It is asserted that the addition of guaiacol renders the injection less painful. The dilution of glycerin should not vary much from 5 parts of water to 21 of glycerin because of the slight solubility (1 : 60) of guaiacol in water.

**Scopolamine Solutions, Deterioration of, in Vials.** H. Lenger. (*Giorn. farm. chim.*, 1913, **62**, 174-5 ; *Chem. Abstr.*, 1915, **9**, 1367.) Two solutions, one of 0.10 per cent. scopolamine-HBr in Ringer solution and the other of 0.03 per cent. pure scopolamine in 0.001 per cent. of N/HBr solution (in order to neutralize the alkalinity of the glass vials), were prepared and distributed in 1 c.c. vials. The activity of the first solution was diminished by  $\frac{2}{3}$  after 5 months, while the activity of the second solution was decreased by  $\frac{1}{4}$  after 9 months. Solutions of scopolamine should, therefore, only be prepared as required.

**Scopolamine Hydrobromide Solutions, Stable.** W. Staub. (*Pharm. Zeit.*, 1914, **59**, 263.) The addition of a small amount of mannitol renders dilute solutions of scopolamine hydrobromide quite stable. The following is a typical formula : Scopolamine hydrobromide, 20 centigrammes ; mannitol, 100 Gm. ; distilled water to 1 litre. Dissolve and sterilize.

**Senna Leaves free from Resin.** Ruediger. (*Zeit. allg. æster. Apoth. Ver. ; Drugg. Circ.*, 1914, **58**, 527.) Senna leaves with 3 parts, by weight, of strong EtOH are macerated for three days with occasional shaking. The mixture is filtered, the leaves are pressed and again allowed to macerate with 2 parts of strong alcohol for 24 hours. They are then deprived of the EtOH by filtration and dried at moderate heat. Leaves thus treated possess a beautiful green colour. The residue left on

evaporating the EtOH solution may be used for veterinary purposes.

**Solidified Liniment.** (*Med. Drugg.*, 1915, 49.) The following formulæ are recommended for the production of a useful household remedy: (No. 1) Oil of origanum, 10; oil of sassafras, 10; oil of turpentine, 10; camphor, 10; oleoresin of capsicum, 5; fluid extract aconite, 4; petrolatum, 30; white wax, 21; alkanet root, q.s. (No. 2) Oil of origanum; oil of sassafras; oil of turpentine; camphor, aa 45; oil of lavender, 10; fluid extract aconite root, 14; oleoresin of capsicum, 16; lanolin; white wax; petrolatum, aa 90; alkanet root, q.s. (No. 3) Oil of origanum; oil of sassafras; oil of turpentine; camphor, aa 10; oil of cajuput, 3; allyl-isothiocyanate, 1; fluid extract aconite root, 5; oleoresin of capsicum, 5; petrolatum, 26; white wax, 20; alkanet root, q.s. Heat the petrolatum and suspend in it the alkanet root, previously bruised, and digest until the desired colour is obtained. Bear in mind that the petrolatum is to be diluted, so the colour should be made strong enough to stand this dilution. Add the white wax and when melted remove from fire and when slightly above the congealing point add the fluid extract and stir rapidly. Add the oils in which the camphor has been dissolved previously, and after mixing add the oleoresin and again mix, this time thoroughly. The amount of capsicum in the liniment is sometimes too strong for a tender skin; they should then be reduced, or the liniment should be diluted with petrolatum.

**Syrup of Ferrous Iodide, Use of Preservatives in.** G. M. Beringer. (*Amer. J. Pharm.*, 1914, 86, 358.) If syrup of iron iodide is carefully made and with the proper amount of sugar, no preservative whatever is needed. However, to overcome careless manipulation on the part of some druggists, it has been deemed advisable to add a preservative. Hypophosphorous acid has the advantage of a reducing value which is not possessed by citric and tartaric acids suggested for this purpose. It has, however, the disadvantage that in the strength directed in the U.S.P., 20 c.c. to 1,000 Gm. of syrup, it will act upon sugar in strong solutions and darken the syrup. This could be overcome by substituting glycerin for a portion of the sugar directed in the formula.

**Tablet Industry, Its Evolution and Present Status.** L. F. Kebler. (*J. Amer. Pharm. Assoc.*, 1914, 3, 848, 939, 1062).

An exhaustive illustrated treatise on tablet making. The author deals with the history of the subject; the apparatus and machinery used; the ingredients employed; the manipulative details requisite; and the analytical methods for checking the amount of active ingredients in the finished tablets. A large number of tabular statements of great length are included in the communication, which occupies over 50 pages of the publication.

**Tablets of Morphine Hydrochloride, Bromide, Compound, Phenacetin Compound, and Cascara Sagrada, Examination of.** J. Herzog. (*Apoth. Ztg.*, 1915, 36, 19, 24; *Chem. Abstr.*, 1915, 9, 1659.) *Morphine hydrochloride tablets.*—Triturate 20 tablets in a mortar and wash the powder quantitatively into a flask with hot EtOH. Extract repeatedly with boiling absolute EtOH, filter and evaporate to dryness. Dissolve the residue in about 5 c.c. hot water and transfer quantitatively by means of an additional 5 c.c. of water to a 100 c.c. glass-stoppered Erlenmeyer, add 3 drops 10 per cent.  $\text{NH}_4\text{OH}$ , shake vigorously for 10 minutes and set aside for 12 hours. Pass the liquid through a small, smooth suction filter, allowing the flask to drain completely, then dry both flask and filter at  $100^\circ\text{C}$ . Dissolve the morphine crystals in 10 c.c.  $\text{N}/10$   $\text{HCl}$ , pour the solution into a 200 c.c. flask, carefully rinsing with water the filter and glass vessels used, then increase the volume of the liquid to about 100 c.c. and titrate under a layer of  $\text{Et}_2\text{O}$  with  $\text{N}/10$   $\text{KOH}$ , using iodoeosin as indicator. *Compound bromide tablets.*—After weighing several tablets, dissolve one in water in a 250 c.c. tared flask, fill to mark and apply a portion of the liquid to tests for sulphates and carbonates. In a 50 c.c. aliquot determine the Br volumetrically in accordance with the Ph.G.V. procedure, using  $\text{K}_2\text{CrO}_4$  as indicator. In a second aliquot determine the halogen gravimetrically by precipitating with  $\text{AgNO}_3$ . *Compound phenacetin tablets.*—Weigh out an amount of the powdered sample equal to 4 tablets and extract repeatedly with boiling absolute EtOH, receiving the solvent in a capsule dish and evaporating to dryness. After disintegrating the residue with a glass rod, moisten with very little water, then add 30 Gm. of water and stir for some time. Transfer the mixture to a small, smooth filter, washing with four 5 c.c. portions of water. Dissolve the residual phenacetin remaining in the dish and on the filter in hot EtOH. Evaporate to dryness and weigh as phenacetin (major portion). Transfer the aqueous liquid to a separator, render slightly acid and extract with about 30 c.c. of  $\text{Et}_2\text{O}$ . The  $\text{Et}_2\text{O}$  solution

yields on evaporation an additional quantity (minor portion) of phenacetin. After rendering the aqueous liquid alkaline extract repeatedly with  $\text{CHCl}_3$  in order to recover the caffeine. *Cascara tablets*.—Heat 1 tablet in 100 c.c. of water until the extractives are dissolved and the inert excipients subside. Pass through a filter and extract 10 c.c. in a test tube with 10 c.c. of  $\text{Et}_2\text{O}$  for 2 minutes. Pour off 3 c.c. of the clear (should be intensely yellow) supernatant liquid into a second test tube and add an equal volume of 5 per cent.  $\text{AmOH}$ , shaking the mixture thoroughly, whereupon a deep coral-red colour should appear. Dilute 2 parts of the original aqueous solution with 18 parts of water and treat 10 c.c. of the resulting liquid in exactly the same manner as above, whereupon the  $\text{Et}_2\text{O}$  solution should be faintly yellow and the ammoniacal solution distinctly coral-red after shaking.

**Tonga and Salicylates, Elixir of.** N. Finkelstein. (*Drugg. Circ.*, 1915, 59, 93.) Tonga, in a 40 powder, 8 oz. 340 grains; cimicifuga, in a 40 powder, 2 oz. 149 grains. Moisten the tonga and the cimicifuga with 3 oz. of a menstruum composed of 18 oz. of alcohol and 48 oz. of distilled water. Allow the moistened drugs to stand in the percolator, without packing, during 6 hours. Then pack firmly and add a sufficient portion of the menstruum to saturate the powder and leave a stratum above it. Then allow the drugs to macerate 6 hours; after which percolate until all of the menstruum has been used. In the percolate dissolve: Sodium salicylate, 4 oz. 170 grains; pilocarpine salicylate, 5 grains; colchicine salicylate, 1 grain; sugar, 21 oz.; and add glycerin, 4 oz.; thoroughly triturate compound spirit of orange, 6 drachms, with purified talcum, 2 oz., and gradually incorporate the other fluid. Filter through moistened paper, and after all the fluid has run through, wash the paper and the residue thereon with enough distilled water to make the filtered liquid measure 64 fl. oz.

## PHARMACOPŒIA REVISION NOTES

**Aether, B.P.** D. B. D o t t. (*Chem. & Drugg.*, 1915, 86, 260.) The monograph on ether in the new B.P. is a distinct advance on that in the B.P. 1898. There are two grades of ether, but both have the sp.g. of 0.720. There is no such anomaly as ether of sp.g. 0.735, which has necessarily the properties of a mixture of  $\text{Et}_2\text{O}$  and  $\text{EtOH}$ , and the use of which led to mistakes in regard

to the solubilities of resins and other substances. Not only have both grades the sp g. 0.720, but both may be made from industrial methylated spirit, provided they comply with the stated tests. It is in regard to these tests that there is room for some criticism. In the first place, the b.p. in both cases is required to be  $34^{\circ}$ – $36^{\circ}\text{C}$ . It is well known that ether prepared with methylated spirit yields a proportion distilling under  $34^{\circ}\text{C}$ , due to the  $\text{Me}_2\text{O}$  formed from the  $\text{MeOH}$ . In the case of the ordinary quality, at least, the test might very well have read: "Boiling-point  $34^{\circ}$ – $36^{\circ}\text{C}$ ., but a proportion may distil below  $30^{\circ}\text{C}$ . if the lower point is due to the presence of  $\text{Me}_2\text{O}$ ." As a general solvent and for local anæsthesia there is no objection to  $\text{Me}_2\text{O}$ . Indeed, it may be doubted whether so small an amount as may possibly be present would be a disadvantage in any of the medical or pharmaceutical applications of  $\text{Et}_2\text{O}$ . But even if we concede that "purified ether" may be expected to boil at  $34^{\circ}$ – $36^{\circ}\text{C}$ ., "ether" might have been allowed to pass with the recognized properties of ether made from methylated spirit. The next point to be considered is the test for "methyl compounds." This applies only to "purified ether." The test consists in shaking the sample of ether with alcohol and water, separating the lower layer, adding  $\text{KMnO}_4$  and  $\text{H}_2\text{SO}_4$ ; then after three minutes adding  $\text{H}_2\text{C}_2\text{O}_4$  and more  $\text{H}_2\text{SO}_4$ , together with decolorized fuchsine solution, all of course in measured proportions. There must be "no violet colour" produced during 20 minutes. The test is all right if by "violet colour" a slight fuchsine tint is intended, and that is probably what is really meant. This is confirmed by the fact that the addition of the least quantity of formaldehyde to ether causes a marked violet coloration with the test. Unfortunately, however, highly purified ether from methylated spirit, and even ether from rectified alcohol, gives generally a faint indigo-blue colour before 20 minutes, and this slight tint has been mistaken for violet within the meaning of the official test. It ought to have been made clear that this was not intended. The author does not agree with the statement of Finemore as to the danger of superheating during distillation. The water-bath, properly used, gives better results than any sort of flame. Among the sources of appreciable error, the adhesion of liquids to glass might have been stated. We were taught to put pieces of metallic foil in the flask, in order that the liquid might boil as readily as in a metal flask, which is the standard apparatus for the purpose.

**Ash Limit for Drugs, Proposed for the U.S.P. IX.** M. I. Wilbert. (*Amer. J. Pharm.*, 1914, 86, 456.) The figures proposed as the limit in the next U.S.P., those accepted in contemporary pharmacopœias, and the amounts given in current literature are compared.

Drug.	U.S.P. IX.	Ph.G. V.	Ph. Austr. VIII.	Ph. Helvet. IV.	Ph Neder. IV	Current Literature.
Acacia . . . .	4	5	3	4	4	1.23 3.64
Aconitum . . . .	6	—	—	6	—	3.6 —6
Agar-agar . . . .	5	—	—	—	—	2.6 —4
Aloe . . . . .	4	1.5	1	1.5	1.5	0.65—5.65
Althea . . . . .	8	—	6	6	3 7	4.6 — 7.3
Amyg. dulc. . . .	4	—	—	—	—	—
Amylum . . . . .	0.5	1	—	0.5	1	0.05—0.27
Anisum . . . . .	10	10	10	10	—	5.6 —18.6
Apocynum . . . .	9	—	—	—	—	3.4
Arnica . . . . .	9	—	8	—	—	6.2 11.5
Asafetida . . . .	15	15	10	20	10	5 —63.75
Aspidium . . . .	1	—	3	—	—	2.2 —5.44
Aurantii Amari Cortex	7	—	6	7	—	3.7 — 5.5
Belladonnae Folia	20	15	15	15	—	2.35—23.5
"    Radix . .	7	—	6	7	—	6.07— 7.84
Benzoinum . . . .	2	2	2	1.5	2	1.2 — 3.8
Buchu . . . . .	4	—	—	—	—	4.2 — 5.8
Calumba . . . . .	8	—	6	8	—	4.8 — 8.2
Cambogia . . . .	2	—	—	—	—	0.5 —18
Cannabis . . . . .	15	—	15	—	—	6.0 14.4
Cantharis . . . .	9	8	—	8	9	6.5 —10.29
Capsicum . . . . .	1*	6.5	6.5	6.5	—	4.3 — 6.6
Cardamomi Semina .	8	—	10	8	8	3.7 — 9.2
Carum . . . . .	8	—	7	8	—	5 —11
Caryophyllus . . .	0.5*	8	8	7	6	5.4 — 7.3
Cimicifuga . . . .	10	—	—	—	—	4.87— 9.65
Cinnamomum Saigoni- cum . . . . .	2*	5	—	5	8	1.3 — 5.6
Cinnamomum zeylani- cum . . . . .	2*	—	5	5	—	3.3 — 6.8
Coccus . . . . .	6	—	—	6	—	3.28— 9.41
Colchici Semen . .	8	—	8	—	—	2.4 — 3.5
Colocynthis . . . .	15	—	7	—	—	3.6 —13.03
Condurango . . . .	12	—	—	—	—	5.26—16.71
Coriandrum . . . .	7.5	—	7	—	—	4.55— 8.1
Cubeba . . . . .	8	8	—	8	10	—
Digitalis . . . . .	10	—	10	10	—	4.6 —18.5
Ergota . . . . .	5	—	—	5	5	2.6 — 4.3
Foeniculum . . . .	10	10	10	10	—	7.0 —23.75
Frangula . . . . .	6	—	5	—	—	3.7 — 6.5
Gambir (Catechu) .	9	6	5	5	5	3.03—32.0
Gentiana . . . . .	6	—	5	6	2 6	2.5 — 5.42
Glycyrrhiza . . . .	7	—	6	6	6	4.4 — 8.96
Granatum . . . . .	16	—	10	15.5	15	3.63—16.60

\* Ash insoluble in dilute HCl.

Drug.	U S P IX	Ph G V.	Ph. Austr VIII	Ph. Helvet. IV	Ph. Neder. IV	Current Literature.
Guaiacum . . . .	4	—	1	1.5	—	1.63-11.7
Humulus . . . .	8	—	—	—	—	9.70-10.13
Hyoscyamus . . . .	30	24	20	—	—	17.39-31.54
Ipecacuanha . . . .	1.8-4.5	—	6	1.8-4	1.8-6	2.83- 9.34
Jalapa . . . . .	6.5	6.5	5	6.5	—	3.2 - 7.55
Kino . . . . .	3	—	—	2	—	1.47- 5.9
Krameria . . . .	5	—	5	—	—	1.4 - 4.46
Lactucarium . . . .	10	—	—	—	10 *	4.91- 6.9
Linum . . . . .	6	5	5	5	—	3.3 - 5.85
Lobelia . . . . .	8	—	8	—	—	5.1 -11.65
Lupulinum . . . .	16	—	10	10	6	6.60-38.25
Lycopodium . . . .	3	3	3	3	5	1 - 4.10
Matricaria . . . .	13	—	13	—	—	12.4-14.8
Moschus . . . . .	8	—	—	8	—	—
Myristica . . . .	5	—	3	5	—	1.23- 3.3
Myrrha . . . . .	8.5	7	6	6	5	4.1 -15.65
Nux Vomica . . . .	3.5	3	—	3.5	3	1.25- 3.62
Physostigma . . . .	7	—	—	—	—	—
Piper . . . . .	2*	—	5	—	—	0.12*-8.3
Podophyllum . . . .	3	—	—	—	—	3.6 - 5.36
Pyrethrum . . . .	5	—	—	—	—	4.7 - 7.5
Rhamnus Purshiana	8	—	6	—	10	4.14- 8.7
Rheum . . . . .	13	12	12	13	—	7.1 -12.12
Santalum Rubrum . .	3	—	5	—	—	—
Sarsaparilla . . . .	10	—	8	—	—	3.6 -34.57
Sassafras . . . . .	30	—	—	—	—	27.85-13.38
Scilla . . . . .	8	5	8	5	—	1.8 - 4.2
Senega . . . . .	5	—	5	—	—	2.6 - 5.7
Senna . . . . .	12	12	10	10	6-8	8.2 -14.32
Senna . . . . .	3*	—	—	—	—	—
Sinapis Alba . . . .	9	—	5	5	8	5.2 - 8.06
Spigelia . . . . .	10	—	—	—	—	7.93-40.81
Stillingia . . . . .	5	—	—	—	—	5.42- 6.85
Stramonium . . . .	20	20	20	—	—	5.8 -22.16
Strophanthus . . . .	5	—	5	—	—	3.8 - 4.8
Taraxacum . . . . .	10	—	8	—	—	5.42-14.8
Tragacantha . . . .	3.5	3.5	—	3.5	3.5	1.88-29
Triticum . . . . .	3	—	3	—	—	0.96- 3.31
Uva Ursi . . . . .	—	—	4	—	—	2.1 - 7.01
Valeriana . . . . .	20	—	10	12	—	6.8 -32.73
Vanilla . . . . .	6	—	10	12	—	—
Zingiber . . . . .	8	7	5	7	8	3.10- 7.9

\* Ash insoluble in dilute HCl.

**Boiling Points, Determination of, Directions for, in B.P. 1914.**  
H. Finne more. (*Pharm. J.*, 1914 [4], 39, 564.) The official directions are adversely criticized. Instructions for manipulation are given which will ensure more accurate results, and, in particular, avoid error from superheating.

**B.P., 1914, Alkaloids,  $\text{CHCl}_3$  and Opium Preparations of.** D. B. DOTT. (*Pharm. J.*, 1915 [4], 40, 206.) Criticisms are made on the official monographs on apomorphine hydrochloride, atropine and atropine sulphate, codeine, diamorphine hydrochloride, morphine and its salts, pelletierine tannate, pilocarpine nitrate, and quinine salts. The solubility of strychnine hydrochloride in water is 1 : 38 not 1 : 60. The morphinometric methods for opium and its tincture are commented on. The amount of free acid in liquor morphinæ hydrochlor. is still excessive and the quantity of EtOH is needlessly large. Three official solutions of morphine, the acetate, hydrochloride and tartrate are not all necessary. The lowering of the sp.g. of  $\text{CHCl}_3$  is not approved. The amount of EtOH indicated by the sp.g. 1.490 is sufficient for preservative purposes. The statement that the B.P. 1914  $\text{CHCl}_3$  does not begin to boil below  $60^\circ\text{C}$ . is incorrect. Appreciable quantities will distil between  $58^\circ$ – $60^\circ\text{C}$ . A limit for the higher b.p. should be given.

**B.P. 1914 ; Solubility of Citric Acid in  $\text{Et}_2\text{O}$ .** J. TAIT. (*Pharm. J.*, 1915 [4], 50, 244.) The statement that citric acid is only slightly soluble in  $\text{Et}_2\text{O}$  is incorrect. It is soluble 1 : 40 in that liquid. Tartaric acid is less soluble. Incidentally, the author remarks that he was unable to obtain commercially either *Aether* or *Aether purificatus* B.P. 1914.

**B.P. 1914, Criticisms on.** T. STEPHENSON. (*Pharm. J.*, 1915 [4], 40, 236.) The term "mil" as the contraction of millilitre is regarded as vulgar slang. Many of the substitutes for official drugs, now given recognition for employment in India, are stated to be more difficult to procure in the bazaars of that country than the drugs they are supposed to substitute. Betel leaves, which receive official recognition, are used in India solely as a masticatory. The official abbreviation "Tr" might stand in some cases, as, for instance, in "Tr. catechu" for either tinctura or trochiscus.

**B.P. 1914, Criticisms on.** C. W. KEMSEY BOURNE. (*Pharm. J.*, 1915 [4], 40, 521.) The B.P. and the B.P.C. make no reference to the odour of acetyl-salicylic acid. The author finds that it, and its preparations, have the odour of free acetic acid. Aspirin is odourless. Comments are also made on the monographs on picric acid ; ammonium carbonate ; lime water ; powdered gentian (for which Bell's test (*Y.B.*, 1909, 108) is



favourably mentioned) ; powdered squill ; and syrup of iodide of iron. The fact that  $\text{ZnO}$  will absorb  $\text{CO}_2$  from the air unless properly stored is noted. The difference in consistence of confection of senna as prepared by several wholesale firms is alluded to. The fact that some makes of gall and opium ointment become mouldy on keeping is attributed to the use of water for rubbing down the powdered opium in order to obtain a smoother product.

**B.P. 1914, Criticisms on.** J. A. Forret. (*Pharm. J.*, 1915 [4], 40, 246.) The question is raised as to how, when a prescription is written in the metric system, the doses will be prescribed. If in the familiar spoonfuls, this will be necessary to translate the quantities into the Imperial measures. In the directions for preparing tinctures, complete saturation of the drug with the menstruum previous to packing should be ordered instead of moistening, packing, and then saturating. Other criticisms are made.

**B.P. 1914, Criticisms of.** D. Murray. (*Pharm. J.*, 1915 [4], 40, 244.) *Acetum scillae*.—The change of strength is not approved and the preparation is considered to be unsatisfactory. After macerating for a week the squills swell and absorb most of the menstruum. After pressing, the acetum is loaded with mucilage, making it extremely difficult to filter. Even after getting it clear, in a day or two it begins to deposit. *Collodium vesicans*.—The cochineal should be macerated in the acetone, and the solution filtered, before adding it to the other ingredients. It will not settle out. *Tinct. capsici*.—The change of alcoholic strength from 70 to 60 per cent. is undesirable. The lower strength of alcohol fails to keep the oleoresin of capsicum in solution. *Ung. acid. boric*.—The additional 3 per cent. of white beeswax makes the ointment too brittle and unworkable in winter. *Ung. potassii iodid*.—The use of a lanolin basis instead of lard is advocated.

**B.P. 1914, Criticisms on.** J. R. Hill. (*Pharm. J.*, 1915, [4], 40, 245. Comments are made on the Preface ; the tests for citric acid ; for purified alum, and purified borax ; the storing of medicinal leaves ; the solutions of ammonium acetate and citrate, and of bismuth ammonio-citrate ; compound tincture of cardamoms ; zinc acetate and volumetric solutions.

**B.P. 1914, Criticisms on.** J. F. Tocher. (*Pharm. J.*, 1915 [4], 40, 207.) The dual semi-equivalent use of the Imperial and metric systems for doses is not approved. The methods and limits for detection and determination of minute traces of Pb and As are commended. Attention is, however, directed to the difficulty in obtaining agreement among different workers with quantitative colour tests due to idiosyncratic divergences of the function of colour vision and appreciation.

**B.P. 1914, Materia Medica of.** H. Stout. (*Pharm. J.*, 1915 [4], 40, 244.) The exclusion of physiological standards for potent drugs is approved. Belladonna leaves are required to be standardized, but the root is not. Hyoscyamus leaves should be standardized. *Podophyllum emodi* is said to yield a resin answering the chemical tests for podophyllin; but it is reputed to be only half as active as the resin of *P. peltatum* rhizome. Although there is now no official distinction between Barbados and Socotrine aloes, these drugs differ markedly in odour, and galenicals prepared from them differ in this respect. A test to detect the unofficial Cape aloes should be given.

**B.P. 1914, Criticisms on.** F. McDiarmid. (*Pharm. J.*, 1915 [4], 40, 237.) The author summarizes and discusses various changes and processes. Alkaloidal extracts should be diluted, when necessary, with the powdered drug, making due allowance for the alkaloidal strength of the latter; not with  $\text{Ca}_3\text{P}_2\text{O}_4$ .

**B.P. 1914, Criticisms on.** J. H. Franklin. (*Pharm. J.*, 1915 [4], 40, 385.) The galenical preparations are dealt with. The use of EtOH 40 per cent. instead of water for making liquid extract of ergot is recommended. For syrup of iodide of iron, the solution of  $\text{FeI}_2$  should be filtered into the mixture of glucose and syrup. A little  $\text{HPH}_2\text{O}_2$  should be allowed as a reducing agent. The great increase in strength in tincture of strophanthus is condemned. It is pointed out that tincture of nux vomica is only half its former strength, yet the dose remains the same. Formulæ for widely popular preparations, such as cod liver oil emulsion, petroleum emulsion, extract of malt with cod liver oil, chemical food, and compound syrup of hypophosphites, should be introduced, as standards.

**B.P. 1914, Criticisms on.** M. M. Irvine. (*Pharm. J.*, 1915 [4], 40, 318.) The comments deal with the metric weights and measures; alternative measures; metric prescribing;

descriptions and formulæ; omissions; inferior drugs; and the list of official abbreviations.

**B.P. 1914, Chemical Nomenclature of.** W. B. Cowie. (*Pharm. J.*, 1915 [4], 40, 236.) The latinity of some of the Latin synonyms is adversely criticized, and changes advocated.

**B.P. 1914, Analytical Chemistry of.** P. A. W. Self. (*Pharm. J.*, 1915 [4], 40, 334, 418.) Comments on and criticisms of the general method of titration in alkaloid assays, and detailed criticism of methods of assay or tests for the following drugs and preparations: Caffeine citrate; cinchona; ipecacuanha; opium; glacial acetic acid; chloral hydrate; copper sulphate; ethyl chloride; the soaps; anhydrous sodium arsenate; and sodium nitrite. The various appendixes are also criticized.

**Cinnamon Bark Oil, B.P. Characters of.** (*Perfumery Record*, 1915, 6, 93.) Difficulty is experienced in obtaining cinnamon bark oil having the solubility 1:4 in EtOH 70 per cent. prescribed by the B.P. Oils of light specific gravity distilled in England, though possessing a very fragrant odour, are usually less soluble than the heavy oils which have been until recently imported from the Continent and which have been suspected to contain synthetic cinnamic aldehyde. The oils distilled in Ceylon generally contain some leaf oil, and require special treatment to purify them, whilst the cinnamon bark oil imported from Seychelles is of low aldehyde content, and is of a very insoluble type. Even by fractionation it is impossible to obtain a soluble product, and for medicinal purposes it must be regarded as quite unsuitable. The Seychelles oil has a specific gravity of 0.936–0.945, an aldehyde content of 22–30 per cent., and refractive index of 1.518–1.520.

**Digestive Ferments and Animal Products, Comparison of the Official Methods for Testing, in Pharmacopœias of Different Countries.** H. T. Graber. (*J. Amer. Pharm. Assoc.*, 1915, 4, 686.) The tests of the different pharmacopœias are compared in a series of tables. The subjects dealt with are Pepsin, Pancreatin, Oxgall, Thyroid Gland, and Peptone, the latter official in the French Codex only.

**Drugs, Unofficial (in U.S.P.), Standards for.** (*J. Amer. Pharm. Assoc.*, 1914, 3, 873, 1597; 1915, 4, 632, 751.) Descriptive monographs, generally including macro- and microscopical mor-

pholical characters, are given for the following drugs : *Agaricus* ; *Albumen Ovi Recens* ; *Asclepias* ; *Baptisia* ; *Delphinium* ; *Dioscorea* ; *Fraxinus* ; *Fructus Rubi* ; *Fructus Rubi Idaei* ; *Fructus Solani Carolinensis* ; *Gemmae Populi* ; *Juglans* ; *Juniperus* ; *Lac Vaccinum* ; *Menyanthes* ; *Oleum Aurantii Amari Corticis* ; *Oleum Aurantii Florum* ; *Oleum Bergamottae* ; *Oleum Myrciae* ; *Ovum Gallinum* ; *Passiflora* ; *Pumex* ; *Sambucus (Flores)* ; *Senecio* ; *Strontii Carbonas* ; *Succus Citri* ; *Succus Pomorum* ; *Trifolium* ; *Trillium* ; *Verbena* ; *Vitelli Ovi Recens*. Chemical or physical tests for these include the following : *Fraxinus*.—White ash bark. Ash not over 10 per cent. *Fructus Solani Carolinensis*.—Horse-nettle berry. Ash about 5 per cent. *Menyanthes*.—Ash not more than 10 per cent. *Oleum Aurantii Amari Corticis*.—Soluble 1 : 4 in EtOH 91 per cent. the solution being neutral to litmus. Soluble in all proportions of absolute EtOH and in glacial  $\text{HC}_2\text{H}_3\text{O}_2$ . Sp g. 0.846 to 0.854 at 25°C. ;  $\alpha_{\text{D}^{25}}$  +92° to 94°. The  $\alpha_{\text{D}}$  of first 10 per cent. fraction, distilled in a 3-bulb flask, should be equal to or slightly higher than that of the original oil. *Oleum Aurantii Florum*.—Sp g. 0.868 to 0.875 at 25°C. ;  $\alpha_{\text{D}^{25}}$  1° 30' to 5°. Shaken with a concentrated solution of  $\text{NaHSO}_4$  it assumes a permanent purple-red colour. Soluble 1 : 1 in EtOH 95 per cent., the solution having a violet fluorescence and a neutral reaction to litmus paper. Soluble 1 : 2 in 80 per cent., becoming cloudy with more. *Oleum Bergamottae*.—Sp.g. 0.875 to 0.880 at 25°C. ;  $\alpha_{\text{D}^{25}}$  + 8° to +24°. Soluble 2 : 1 in EtOH, should give a clear solution, not becoming turbid with more. Soluble 1 : 2 in 80 per cent. EtOH with not more than a slight cloudiness and no separation of globules. Soluble in all proportions in glacial  $\text{HC}_2\text{H}_3\text{O}_2$ . If about 2 Gm. of the oil be evaporated on a water-bath, until the odour has completely disappeared, a soft, green, homogeneous residue should be left, amounting to not more than 6 per cent. of the oil (fixed oils). To 2 Gm. of the oil add 10 c.c. of alcoholic N/KOH, evaporate to dryness and incinerate. Extract the ash with water and acidify with dilute  $\text{HNO}_3$ , no cloudiness should be produced on addition of  $\text{AgNO}_3$  reagent (Chlorinated Compounds). To 2 Gm. of the oil add 20 c.c. of N/2 KOH and heat the mixture in a flask on a water-bath under a reflux condenser for half an hour. Cool the mixture and add 100 c.c. of distilled water and titrate with N/2  $\text{H}_2\text{SO}_4$  with phenolphthalein indicator, not more than 12.6 c.c. of acid should be required, indicating a minimum content of 36 per cent. of ester, calculated

as linalyl acetate, in the oil. *Oleum Myrciae*.—Sp.g. 0.965 to 0.985 at 25°C.;  $\alpha_{25} - 2^\circ$  to  $-6^\circ$ . With an equal volume of EtOH, glacial  $\text{HC}_2\text{H}_3\text{O}_2$ , or  $\text{CS}_2$ , it yields slightly turbid solutions. The EtOH solution is slightly acid to litmus paper. When mixed with an equal volume of a concentrated solution of NaOH it forms a semi-solid mass. If 2 drops of the oil be dissolved in 4 c.c. of EtOH 95 per cent., and a drop of  $\text{Fe}_2\text{Cl}_6$  reagent be added, a light green colour will be produced; and if the same test be made with a drop of diluted  $\text{Fe}_2\text{Cl}_6$  reagent, prepared by diluting the test solution with four times its volume of water, a light bluish coloration will be produced, which soon disappears. To 3 drops of the oil, contained in a small test-tube, add 3 drops of  $\text{H}_2\text{SO}_4$ , cork the test-tube and allow the mixture to stand for half an hour, a resinous mass should be obtained. On adding to this mass 4 c.c. of diluted EtOH 49 per cent., vigorously shaking the mixture, and gradually heating on a water-bath to the boiling point, the liquid should remain nearly colourless, and should not acquire a red or purplish-red colour (distinction from oil of pimento and oil of cloves). Shake 1 c.c. of the oil with 20 c.c. of hot water, the water should not give more than a scarcely perceptible acid reaction with litmus paper. If, after cooling, the liquid in the test above be passed through a wet filter, the clear filtrate should produce, with a drop of  $\text{Fe}_2\text{Cl}_6$ , only a fugitive greyish green, and not a blue or violet colour should be given (absence of phenol). *Passiflora*.—Ash not over 12 per cent. *Pumex*.—When 10 Gm. of powdered pumice is boiled for half an hour with 50 c.c. of distilled water, keeping up the quantity of liquid, and then filtered, the filtrate should be neutral to litmus. Half of it evaporated and dried at 110°C. should not yield more than 0.01 Gm. of residue. The remaining half after slightly acidifying with HCl should not give a blue colour with  $\text{K}_4\text{Fe}(\text{CN})_6$ . On boiling 1 Gm. with 25 c.c. of dilute HCl for about half an hour, maintaining the liquid at approximately the same volume, and then filtering, the residue on evaporating to dryness and filtering should not exceed 0.05 Gm. *Sambucus*.—Ash not to exceed 8 per cent. and should be white. *Strontii Carbonas*.—1 Gm. should yield a clear solution with 10 c.c. of diluted HCl (absence of sulphate). 10 c.c. of a 1 : 100 solution, dissolved with a slight excess of  $\text{HNO}_3$  added to the water, should not become more than slightly opalescent at once on adding  $\text{AgNO}_3$  reagent (limit of chloride.) Dissolve 1 Gm. in water by means of a slight excess of acetic acid, and dilute to 100 c.c. On adding to

10 c.c. of this solution 5 drops of  $K_2Cr_2O_7$  reagent no turbidity should develop within 5 minutes (limit of barium). 0.5 Gm. dissolved in diluted HCl should not respond to the U.S.P. time limit test for heavy metals. Shake 2 Gm. with 25 c.c. of water and filter, on evaporation of the filtrate and drying the residue at  $100^\circ C$ . it should weigh not more than 0.01 Gm. (limit of soluble substances). Dissolve about 1.5 Gm. (accurately weighed) in 30 c.c. of N/HCl and titrate the excess of acid with N/NaOH, using methyl orange as indicator. Each c.c. of N/HCl consumed is equivalent to 0.07381 Gm.  $SrCO_3$ . *Succus Citri*.—The expressed juice of the ripe fruit of *Citrus medica*, var. *acida* 100 c.c. should contain from 5 to 10 Gm. of total acids, calculated as crystallized citric acid. Sp.g. 1.025 to 1.040 at  $25^\circ C$ . To 5 c.c. of lime juice add 20 c.c. solution of KOH and heat with 0.5 Gm. of granular Al or Al foil on a water-bath for 10 minutes, no odour of ammonia should be noticeable at any time during the heating (absence of nitrates). If 0.1 c.c. of  $BaCl_2$  reagent be added to 5 c.c. of clear filtered lime juice, only a slight turbidity should be produced after standing two minutes (limit of sulphates). If 0.1 c.c. of  $HNO_3$  followed by 0.1 c.c. of  $AgNO_3$  reagent be added to 5 c.c. clear filtered lime juice, only a slight opalescence should be produced after standing two minutes (limit of chlorides). If 5 c.c. each of  $H_2SO_4$ , EtOH and lime juice be heated, no odour of acetic ether should be developed (limit of acetates). Add 1 c.c. of an aqueous solution of  $K(C_2H_3O_2)$  (1 to 3) to 5 c.c. of filtered lime juice and then add to the mixture alcohol in excess, a slight cloudiness may occur but no crystalline precipitate should be formed within 15 minutes (limit of tartrates). Upon evaporation and ignition until free from carbon, lime juice should not leave more than 0.5 per cent. of ash. The ash from 5 c.c. of lime juice when dissolved in a few drops of  $HNO_3$  and diluted with water should show not more than traces of phosphate when tested with ammonium molybdate reagent. Lime juice should contain not more than 0.04 per cent. of sulphurous acid ( $SO_2$ ) when tested by the method of the U.S.P. IX Revision for determining  $SO_2$  in gelatin. Upon distilling 200 c.c. of lime juice with excess of  $Ca(OH)_2$  until 100 c.c. of distillate is obtained, the specific gravity of the distillate should indicate not more than 2 per cent. of absolute alcohol by volume in the distillate or 1 per cent. in the lime juice. Shake 10 c.c. of lime juice acidified with  $H_2SO_4$  with 25 c.c. of  $Et_2O$ , separate the  $Et_2O$  and evaporate it to dryness, the residue should not be

crystalline and, when dissolved in about 3 c.c. of water, should not produce a purplish colour on addition of one drop of  $\text{Fe}_2\text{Cl}_6$  reagent (salicylic acid). Shake 10 c.c. of lime juice acidified with  $\text{H}_2\text{SO}_4$  with 25 c.c. of  $\text{Et}_2\text{O}$  and separate the ether and evaporate it to dryness, the residue should not be crystalline and, when dissolved in 3 c.c. of water and carefully neutralized with  $\text{AmOH}$ , should not produce a flesh-coloured precipitate on the addition of one drop of  $\text{Fe}_2\text{Cl}_6$  reagent (benzoic acid). Dilute 20 c.c. of lime juice with 100 c.c. of water, filter, if necessary, and add 4 c.c. of diluted  $\text{HCl}$ . Into this solution immerse a piece of wool which has been boiled in a very dilute solution of  $\text{KOH}$  and then washed in water, and boil for 5 to 10 minutes. Remove the wool, wash thoroughly in water, and boil in a very dilute solution of  $\text{HCl}$ . After washing out the acid with water, boil with about 200 c.c. of 2 per cent. solution of  $\text{AmOH}$  until the colour on the wool, if any, is dissolved. Remove the wool, and add a slight excess of  $\text{HCl}$  to the solution. Immerse in this solution another piece of wool which has been treated with  $\text{KOH}$  solution in the same manner as the first. Boil. This second piece of wool should not be dyed (aniline dyes). *Trillium*.—Ash not more than 5 per cent.

**Drugs, Unofficial, Proposed Monographs for N.F.** (*J. Amer. Pharm. Assoc.*, 1914 [3], 1597; 1915 [4], 632, 751.) *Allium*.—Garlic. The botanical and microscopical characters of the bulb of *Allium sativum* are detailed. The drug should be used fresh.

*Flores Verbasci*.—Dried mullein flowers; from *Verbascum phlomoides* or *V. Thapsiforme*. Botanical and physical characters given.

*Hydrangea*.—The dried rhizome of *Hydrangea arborescens*, Seven barks. Macro- and microscopical characters of the whole and powdered drug are given.

*Inula*.—Elecampane. Dried rhizome and roots of *Inula helenium* are described, both whole and powdered; the macro- and microscopical characters of both being detailed.

*Iris*.—Orris. The rhizome of *Iris florentina* and *I. pallida*. Characters of the whole drug only are given. Ash not to exceed 6 per cent.

*Macis*.—Mace. The arillode of the seed of *Myristica fragrans*. Macroscopic characters of the whole drug and micro-appearance of the powder detailed, also those of false Bombay mace. When moistened with  $\text{HCl}$  no greenish colour should be given (difference

from *Myristica malabarica*, Bombay mace). A 1 : 10 EtOH extract, treated with  $K_2CrO_4$  reagent, should give a yellow precipitate not changing to red on standing, nor should the liquid develop a red colour (Bombay mace). A piece of filter paper moistened with the same extract should not give a blood-red colour with 1 drop of KOH solution (Bombay mace). Ash not more than 3 per cent. ; and this should be almost completely soluble in HCl. Mace should yield not less than 8 per cent. of volatile  $Et_2O$  extract ; nor less than 20 per cent., nor more than 30 of non-volatile  $Et_2O$  extract.

*Petroselinum*.—Parsley root, from *Petroselinum sativum*. Macro- and micro-characters of the whole drug and powder are given. Ash not more than 6 per cent.

*Pimpinella*.—Pimpinel root, from *Pimpinella magna*. Macro- and micro-characters of whole and powdered drug are given.

*Potassii Formas*.—To contain when dry not less than 98 per cent. of  $KCOOH$ . It should be kept in well-stoppered bottles. Very deliquescent, colourless crystals, or white crystalline powder, odourless, taste saline bitter ; very soluble in water, soluble in EtOH. Its aqueous solution is slightly alkaline to litmus, but should not redden phenolphthalein. When the salt is heated, H is evolved and a residue is left which effervesces with acid, and imparts to a non-luminous flame a violet colour. On adding  $NaHC_4H_4O_6$  reagent to the aqueous solution of the salt (1 : 20) a white crystalline precipitate is slowly formed which dissolves on the addition of AmOH. When  $Fe_2Cl_6$  reagent is added to the aqueous solution of the salt (1 : 20) a red colour is produced which is discharged by strongly acidifying with  $H_2SO_4$ . The addition of  $HgCl_2$  reagent to the warm aqueous solution of the salt (1 : 20) produces a white precipitate of  $HgCl$  which turns grey on further warming in the presence of an excess of the formate. The aqueous solution of the salt (1 : 100) should comply with the U.S.P. test for limit of heavy metals. Ten c.c. portions of the aqueous solution of the salt (1 : 20) slightly acidified with  $HCl$  should not be rendered turbid within 5 minutes by the addition of  $Am_2C_2O_4$  reagent (calcium), nor by  $CaCl_2$  reagent (oxalic acid). Weigh accurately about 2 Gm. of potassium formate, previously dried to constant weight at  $120^\circ C.$ , and ignite it thoroughly at a temperature not exceeding a red heat. Dissolve the residue in hot distilled water, filter, if necessary, and wash until washings cease to affect phenolphthalein. Then cool the solution and titrate with  $N/H_2SO_4$ , with methyl orange as



indicator. The result should indicate not less than 98 per cent. of  $\text{KCOOH}$ . Each c.c. of  $\text{N}/\text{H}_2\text{SO}_4 = 0.08411$  Gm. of  $\text{KCOOH}$ .

*Quininæ Valeras.*—Should be kept in well-stoppered amber-coloured vials. White lustrous crystals, having an odour of valeric acid and an intensely bitter taste. Very sparingly soluble in cold water, soluble in hot water, becoming less soluble by age on account of loss of valeric acid; readily soluble in  $\text{EtOH}$ . Its aqueous solution is neutral or slightly alkaline to litmus. On treating 10 c.c. of the aqueous solution of the salt (1 : 1,000) with a few drops of  $\text{Br}$  water, then with an excess of  $\text{AmOH}$ , an emerald green colour will be produced. The aqueous solution acidified with  $\text{H}_2\text{SO}_4$  exhibits a blue fluorescence and emits the odour of valeric acid. Ash not to exceed 0.1 per cent. About 0.1 Gm. of the salt should dissolve in 5 c.c. of  $\text{H}_2\text{SO}_4$  without producing more than a light-yellow colour. Ten c.c. portions of the cold saturated aqueous solution of the salt should not give more than a slight turbidity with  $\text{BaCl}_2$  solution when acidulated with  $\text{HCl}$  (sulphate) nor with  $\text{AgNO}_3$  solution when acidified with  $\text{HNO}_3$  (chloride). The quinine obtained by shaking out the salt with  $\text{AmOH}$  and  $\text{CHCl}_3$  should comply with the U.S.P. test for absence of excessive amounts of other cinchona alkaloids.

*Sodii Formas.*—Should contain, when dried, not less than 98 per cent.  $\text{NaCOOH} = 68.01$ . It should respond to the general tests given under potassium formate except those for the alkaline base. It should be kept in well-stoppered bottles. A white, crystalline powder, or colourless crystals, containing one molecule of water; odourless and having a saline bitter taste. It is very soluble in water, sparingly soluble in alcohol. Weigh accurately about 2 Gm. of sodium formate, previously dried to constant weight at  $120^\circ\text{C}$ ., and ignite it thoroughly in a crucible at a temperature not exceeding a red heat. Dissolve the residue in hot distilled water, filter, if necessary, and wash until washings cease to affect phenolphthalein. Then cool the solution and titrate with  $\text{N}/\text{H}_2\text{SO}_4$ , using methyl orange as indicator. The result should indicate not less than 98 per cent. of anhydrous  $\text{NaCOOH}$ . Each c.c. of  $\text{N}/\text{H}_2\text{SO}_4 = 0.06801$  Gm. of anhydrous  $\text{NaCOOH}$ .

*Strychninæ Valeras.*—A white crystalline powder, having an odour of valeric acid and an intensely bitter taste. Sparingly soluble in water, becoming less soluble by age on account of loss of valeric acid; soluble in  $\text{EtOH}$  or  $\text{CHCl}_3$ , slightly soluble in

**Et<sub>2</sub>O.** Its aqueous solution is neutral or slightly alkaline to litmus. On dissolving about 0.05 Gm. of strychnine valerate in 2 c.c. of H<sub>2</sub>SO<sub>4</sub> not more than a faint yellowish colour should be produced; but on adding a fragment of K<sub>2</sub>Cr<sub>2</sub>O<sub>7</sub> a deep violet colour will be produced which changes to orange or yellow. When H<sub>2</sub>SO<sub>4</sub> is added to the salt, the odour of valeric acid is evolved. Ash not to exceed 0.1 per cent. Ten c.c. portions of the aqueous solution of the salt (1 : 100) should not be affected at once by BaCl<sub>2</sub> solution when acidified with HCl (sulphate) nor by AgNO<sub>3</sub> solution when acidified with HNO<sub>3</sub> (chloride). About 0.02 Gm. of the salt moistened with HNO<sub>3</sub> may be coloured yellow, but should not become red or reddish (brucine).

**Thymus.**—Thyme. Dried flowering tops of *Thymus vulgaris*. Macro- and micro-characters given. Ash not to exceed 10 per cent.

**Aluminii Chloridum.**—Contains AlCl<sub>3</sub> + 6H<sub>2</sub>O equivalent to not less than 20.5 per cent. of Al<sub>2</sub>O<sub>3</sub>. White, or yellowish white, deliquescent, crystalline powder; nearly odourless; taste sweet and very astringent. Soluble about 1 : 1 in water and 1 : 3 in EtOH at 25°C., also soluble in glycerin. An aqueous solution (1 in 10), should be clear, and show an acid reaction to litmus, and give the reactions for Al and Cl which are described in detail. Ten c.c. of an aqueous solution (1 in 100), after the addition of 0.2 c.c. of BaCl<sub>2</sub> T. S., must not become cloudy within one minute (limit of sulphate). Ten c.c. of an aqueous solution (1 in 50) must not respond to the time limit test for heavy metals omitting the addition of AmOH. The addition of 0.3 c.c. of K<sub>4</sub>FeCy<sub>6</sub> T. S. to 20 c.c. of an aqueous solution (1 in 150) must not produce a blue coloration within one minute (iron). Five c.c. of an aqueous solution (1 in 25) must not respond to the U.S.P. modified Gutzeit test for As. Dissolve about 0.5 Gm. of the salt, accurately weighed, in 100 c.c. of water, add 1 Gm. of AmCl and then precipitate the Al<sub>2</sub>(OH)<sub>3</sub> by the addition of a slight excess of AmOH to the boiling solution. Collect the precipitate, wash with distilled water, dry, ignite thoroughly and weigh. The weight of the aluminium oxide so obtained must not be less than 20.5 per cent. of the weight of the aluminium chloride used.

**Aluminii Sulphas.**—Should contain not less than 99.5 per cent. of pure aluminium sulphate Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub> + 16H<sub>2</sub>O. A white, crystalline powder, or shining plates, or crystalline fragments; without odour, having a sweetish and afterwards an astringent

taste, and permanent in the air. Soluble in water 1 : 1 at 25°C. (77°F.), more soluble in boiling water, but insoluble in EtOH. When gradually heated to about 200°C. it loses its water of crystallization (45.7 per cent. of its weight). The aqueous solution of the salt has an acid reaction upon blue litmus paper. It should afford the reactions for Al and for  $\text{SO}_4$  which are detailed. If 1 Gm. of aluminium sulphate be gently heated with 5 c.c. of KOH reagent, the liquid should not evolve the odour of  $\text{NH}_3$ . A filtered, aqueous solution of the salt (1 in 10) should not become more than faintly opalescent within five minutes after the addition of an equal volume of N/10 thiosulphate (limit of free acid). The salt should pass the same test as prescribed for the chloride, for As, heavy metals, and Fe. When submitted to a similar gravimetric test to that given for the chloride, the amount of  $\text{Al}_2\text{O}_3$  obtained should be 16.1 per cent. of the weight of the  $\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$  taken.

*Antimonii Oxidum*.—Contains not less than 97 per cent. of  $\text{Sb}_2\text{O}_3$ . A white or greyish white odourless and tasteless powder. Insoluble in water, EtOH or  $\text{HNO}_3$ , readily soluble in HCl without effervescence, and also in a warm solution of  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$  or in a boiling solution of  $\text{KHC}_4\text{H}_4\text{O}_6$ . It gives the reactions for  $\text{Sb}_2\text{O}_3$  which are detailed. The solution of 0.1 Gm. of the oxide in 3 c.c. of HCl and 5 c.c. of distilled water is not rendered turbid at once by a few drops of  $\text{BaCl}_2$  reagent (sulphate). The solution of 0.1 Gm. of the oxide in 10 c.c. of distilled water and 1 Gm. of  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$  does not become more than slightly opalescent after the addition of 0.2 c.c.  $\text{HNO}_3$  and 0.2 c.c.  $\text{AgNO}_3$  reagent (chloride). Dissolve 0.5 Gm. of the oxide in 10 c.c. of HCl, dilute the solution with distilled water until it begins to become permanently turbid and then precipitate with  $\text{H}_2\text{S}$ . This precipitate when thoroughly washed with distilled water dissolves in AmHS or NaHS solution, without leaving a black insoluble residue (copper, lead). A solution of 0.1 Gm. of antimony oxide in 5 c.c. of HCl does not respond to Bettendorf's test for arsenic. Weigh accurately about 0.2 Gm. of antimony oxide, dissolve it by warming with a solution of 1 Gm. of  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$  in 10 c.c. of distilled water (adding a few drops of HCl to aid the solution), nearly neutralize the solution with  $\text{Na}_2\text{CO}_3$ , add 40 c.c. of a cold saturated solution of  $\text{NaHCO}_3$  and titrate at once with N/10 I with starch indicator. The titration should show not less than 97 per cent. of  $\text{Sb}_2\text{O}_3$ . Each c.c. of N/10 I = 0.007210 Gm. of  $\text{Sb}_2\text{O}_3$ .

*Antimonii Sulphidum Purificatum*.—Contains Sb correspond-

ing to not less than 97 per cent. of  $\text{Sb}_2\text{S}_3$ . A heavy odourless and tasteless greyish black powder. Insoluble in water or  $\text{EtOH}$ , soluble in  $\text{HCl}$  with the evolution of  $\text{H}_2\text{S}$ . At a temperature below a red heat it fuses to a dark brown liquid. A solution made by boiling the sulphide with a moderate excess of  $\text{HCl}$ , until the vapours no longer blacken lead acetate paper, yields, when added to about 10 times its volume of water, a white precipitate which is changed to orange by  $\text{H}_2\text{S}$ . Intimately mix 2 Gm. of the sulphide with 8 Gm. of pure  $\text{NaNO}_3$ , fuse the mixture in a porcelain crucible, and after cooling boil the mass with 25 c.c. of distilled water and filter. Acidulate the filtrate with  $\text{HNO}_3$ , boil until no more  $\text{N(O)}$  is evolved, then dissolve in the solution about 0.1 Gm. of  $\text{AgNO}_3$ , filtering again, if necessary, and cautiously overlay 10 c.c. of this solution with a few drops of  $\text{AmOH}$ . Not more than a white cloud, but no red or reddish precipitate appears at the line of contact of the two liquids (limit of  $\text{As}$ ). Weigh accurately about 1 Gm. of purified antimony sulphide, mix it with 20 c.c. of  $\text{HCl}$ , and then with a clear solution of 5 Gm. of  $\text{H}_2\text{C}_4\text{H}_4\text{O}_6$  in 10 c.c. of water and heat the mixture gently on the water-bath until the vapours no longer blacken  $\text{Pb2C}_2\text{H}_3\text{O}_2$  test paper. Filter, wash the residue, if any, until free from acid, then ignite. Its weight should not exceed 1 per cent. Dilute the filtrate and washings to 200 c.c.; nearly neutralize 50 c.c. with  $\text{Na}_2\text{CO}_3$ ; add 40 c.c. of cold-saturated  $\text{NaHCO}_3$  solution and titrate at once with  $\text{N/10 I}$  with starch indicator. Each c.c. of  $\text{N/10 I} = 0.008415$  Gm. of  $\text{Sb}_2\text{S}_3$ . The result should not be below 97 per cent.

*Berberis*.—Oregon grape root. The rhizome and roots of the various species of section *Odostemon* of the genus *Berberis*. Macroscopic characters of the drug and the microscopic appearance of the powder are described.

*Bismuthi Citras*.—Containing  $\text{BiC}_6\text{H}_5\text{O}_7$  equivalent to not less than 55 or more than 59 per cent. of  $\text{Bi}_2\text{O}_3$ . Prepared by heating  $\text{BiONO}_3$  100 and citric acid 75 with water 400 on the boiling water-bath until a drop withdrawn does not precipitate with water. Then add water 500: allow to deposit, collect, wash until tasteless, then dry at gentle heat. Gives the characteristic reactions of  $\text{Bi}$  and citric acid which are detailed. A mixture of 0.01 Gm. of the salt in 1 c.c. of water and 5 c.c. of  $\text{H}_2\text{SO}_4$ , carefully poured over 5 c.c. of  $\text{FeSO}_4$  solution, should show no red or brown zone within 5 minutes (nitrates). Ignite 3 Gm. of the salt. Dissolve the residue in just sufficient  $\text{HNO}_3$ ,

and pour into 100 c.c. of distilled water. Filter out the precipitate, evaporate the filtrate to 30 c.c. on the water-bath, and filter and divide into 5 c.c. portions. These should answer the tests for purity given under *Bismuthi Subcarb.* in U.S.P. IX. Three Gm. of  $\text{Bi}_2\text{C}_3\text{H}_5\text{O}_7$ , after ignition and treatment with  $\text{HNO}_3$ , as directed in the following test, should not respond to the Bettendorf's test for As as stated in the U.S.P. Ignite 1 Gm. of bismuth citrate thoroughly, in a porcelain crucible, and, after cooling, add 5 c.c. of  $\text{HNO}_3$  to the residue, drop by drop, warming until complete solution is effected, then evaporate to dryness, and again ignite it; a residue of  $\text{Bi}_2\text{O}_3$  should be left weighing not less than 0.56 Gm., nor more than 0.58 Gm.

*Brayera* (Cusso).—The dried panicles of the pistillate flowers of *Haegenia abyssinica*. Descriptive botanical monograph given.

*Bromum*.—Should contain not less than 97 per cent. of Br and not more than 3 per cent. of Cl. Bromine should be kept in glass-stoppered bottles in a cool place, the bottle being enclosed in a larger vessel with the space between filled with some compound capable of absorbing and combining with any Br vapours which might be given off. Sp.g. about 3.016 at  $25^\circ\text{C}$ . B.p., about  $63^\circ\text{C}$ . Bromine destroys the colour of solutions of litmus and indigo, and imparts a yellow colour to starch solution. On adding 5 c.c. of Br to an excess of KOH, re-agent it should combine to form a permanently clear liquid without the separation of oily drops (organic bromine compounds). On shaking 10 c.c. of a saturated aqueous solution of Br with a slight excess of reduced iron until it becomes nearly colourless, the filtered liquid, on the addition of 5 drops of  $\text{Fe}_2\text{Cl}_6$  reagent and of 5 drops of starch reagent, should not assume a blue colour (iodine). Dissolve 10 Gm. of KI in 25 c.c. of distilled water, introduce the solution into a 100 c.c. glass-stoppered graduated flask and determine accurately the weight of the flask and its contents. Add about 1 Gm. of Br and determine its exact weight and then fill the flask to the mark with distilled water. The titration of 25 c.c. of this solution with N/10 thiosulphate solution, using starch solution as indicator, and the calculation to the amount of Br originally taken, should show not less than 97 per cent. Each c.c. N/10 thiosulphate used = 0.007992 Gm. of Br and 0.003546 Gm. of Cl.

*Calcii Phosphas Præcipitatus*.—Should contain, when dried to constant weight, not less than 96 per cent. of  $\text{Ca}_3\text{P}_2\text{O}_8$ . Precipitated calcium phosphate occurs as a white, amorphous or

micro-crystalline, odourless and tasteless bulky powder, permanent in the air. At an intense, white heat, the salt fuses without decomposition. Almost insoluble in cold water; partly decomposed by boiling water, which dissolves out the acid salt; almost insoluble in  $\text{HC}_2\text{H}_3\text{O}_2$ , except when freshly precipitated; easily dissolved by  $\text{HCl}$  or  $\text{HNO}_3$ ; insoluble in  $\text{EtOH}$ . When moistened with  $\text{AgNO}_3$  reagent, either before or after ignition, the salt acquires a yellow colour (distinction from acid calcium phosphate which after similar treatment remains white). Suspend 2 Gm. of  $\text{Ca}_3\text{PO}_4$  in 20 c.c. of distilled water; add  $\text{HNO}_3$  drop by drop until solution is effected, then make up to 40 c.c. Portions of this solution should give the characteristic tests for  $\text{Ca}$  and  $\text{PO}_4$  which are detailed. Five c.c. of the solution to which 0.5 c.c. of  $\text{AgNO}_3$  solution is added should not give more than a slight turbidity ( $\text{Cl}$ ). Another 5 c.c. strongly acidified with  $\text{HNO}_3$  to which 1 c.c. of  $\text{K}_2\text{SO}_4$  reagent is added should not give a turbidity in 15 minutes ( $\text{Ba}$ ). An aqueous solution 1:20 obtained by suspending ( $\text{Ca}_3\text{PO}_4$ ) in water and dissolving by adding  $\text{HCl}$  drop by drop, heating until dissolved, should not respond to the time limit test of the U.S.P. for heavy metals. Five c.c. of a 1:25 solution of  $\text{Ca}_3\text{PO}_4$  in dilute  $\text{HCl}$  should give no reaction with the U.S.P. test for  $\text{As}$ .

*Calendula*.—Dried ligulate florets of *Calendula officinalis*. Botanical description given.

*Cassia Fistula*.—The fruit of *Cathartocarpus fistula*. Macroscopical characters given.

*Cataria*.—Catmint, Catnep. Dried leaves and flowering tops of *Nepeta cataria*. Botanical description given. Ash not to exceed 16 per cent.

*Chimaphila*.—Pipsissewa. Dried leaves of *Chimaphila umbellata* with not more than 5 per cent. of stem or other foreign substance. Botanical description is given.

*Chirata*.—Botanical description given.

*Condurango*.—Macroscopical description of the whole bark and micro-structure of the powder given.

*Conium*.—The fully grown unripe fruit botanically described. Conium should be assayed as follows: Conium, in No. 40 powder, 15 Gm.; solution of  $\text{NaOH}$ , 15 c.c.; purified petroleum benzin;  $\text{N}/\text{HCl}$ ;  $\text{Na}_2\text{CO}_3$  reagent;  $\text{N}/10$   $\text{H}_2\text{SO}_4$ ;  $\text{N}/50$   $\text{KOH}$ . Cochineal indicator of each, a sufficient quantity. Place the conium in a 250 c.c. Erlenmeyer flask,

add 150 c.c. of purified petroleum benzin and then 15 c.c. of NaOH solution, insert the stopper securely, and shake the flask vigorously at frequent intervals during 6 hours. Allow the solution to separate and decant 100 c.c. of the clear benzin solution (representing 10 Gm. of the drug) into a separator ; shake this out with successive portions of 20 c.c., 10 c.c., 5 c.c. and 5 c.c. of N/HCl. If a few drops of this last washing gives an alkaloidal reaction with I reagent, continue the shaking out with successive portions of 5 c.c. each of N/HCl until the alkaloid is all extracted. Collect the acid washing and concentrate by evaporation on a water-bath to 10 c.c., cool and transfer the liquid to a separator, then add  $\text{Na}_2\text{CO}_3$  in excess. Extract the alkaloid by shaking out with successive portions of 15 c.c. each of purified petroleum benzin. Separate the benzin washings and filter into a beaker. Then add exactly 10 c.c. of N/10  $\text{H}_2\text{SO}_4$ , and stir thoroughly so as to mix the acid and benzin solutions. Evaporate the benzin in a current of warm air. Then cool, add 5 drops of cochineal solution and titrate the uncombined acid with N/50 KOH solution. Each c.c. of N/10  $\text{H}_2\text{SO}_4$  found to be combined = 0.0426 Gm. of conine. This amount  $\times 10$  gives the percentage of conine in the sample. Average dose — 0.200 Gm. (3 grains).

*Convallaria*.—The dried rhizome. Botanical characters and micro-structure of the whole drug and the histology of the powder given

*Crocus*.—The stigmas of *Crocus sativus*, without admixture of more than 10 per cent. of the yellow styles and other harmless impurities. Macro- and micro-characters given. When placed in  $\text{H}_2\text{SO}_4$  the stigmas should be immediately coloured blue, gradually changing to violet, and finally become a deep wine-red colour. Add 0.010 Gm. of finely powdered saffron to 100 c.c. of cold water, allow it to macerate for several hours and filter ; upon adding 10 c.c. of this filtrate to 100 c.c. of water, it should give a distinct, yellow-coloured solution. Macerate 0.010 Gm. of saffron in 5 c.c. of MeOH ; a deep orange colour should be imparted to the liquid. Macerate 0.010 Gm. of saffron in 5 c.c. of acetone, EtOH 94 per cent., or absolute EtOH ; a distinct, lemon-yellow colour should be produced. With similar quantities of saffron and  $\text{Et}_2\text{O}$  a very light lemon-yellow colour should be produced. With corresponding quantities of saffron and  $\text{CHCl}_3$  a very slight, yellow tinge should be imparted ; and with corresponding quantities of saffron and xylene,  $\text{C}_6\text{H}_6$ ,

$\text{CS}_2$  and  $\text{CCl}_4$  the solution should remain colourless. When placed between filter paper no oily spots should be given. When dried at  $100^\circ\text{C}$ . the loss should not exceed 14 per cent. Ash not to exceed 7.5 per cent., and should not be fusible.

*Cupri Sulphas*.—To contain not less than 63.61 or more than 66.79 per cent. of anhydrous  $\text{CuSO}_4$ . To give the usual reaction for  $\text{CuSO}_4$ . Weigh accurately about 1 Gm. of the salt in un-effloresced crystals, dissolve it in 50 c.c. of water, add 4 c.c. of  $\text{HC}_2\text{H}_3\text{O}_2$  and 3 Gm. of KI. Titrate the liberated I with N/10 hypo, using starch indicator. Each c.c. of N/10 hypo = 0.015964 Gm. of anhydrous  $\text{CuSO}_4$ . The limits should be those given above.

*Cypripedium*.—The dried rhizome of *C. pubescens* or of *C. parviflorum*. Botanical description given.

*Euonymus*.—The bark of *E. atro-purpureus*. Not to have more than 3 per cent. of adhering wood. Botanical description of the whole drug and micro-character of the powder given.

*Eupatorium*.—Bone-set. The dried leaves and flowering tops of *E. perfoliatum*. Botanical description given.

*Extractum Carnis*.—The residue obtained from fresh beef broth by evaporation at low temperature. A yellowish-brown to dark-brown, slightly acid, pasty mass having an agreeable meat-like odour and taste. Twenty-five Gm. of extract of beef diluted to 250 c.c. with distilled water yields a nearly clear solution, free from sediment. Portions of this solution should answer to the following tests: Ten c.c. of the solution boiled for one minute with 1.5 Gm. of purified animal charcoal, the loss by evaporation restored and filtered, the filtrate produces no blue coloration when one drop is added to 3 drops of diphenylamine solution in concentrated  $\text{H}_2\text{SO}_4$  (1 : 100) (limit of nitrates). Ten c.c. of the solution when distributed over sand or asbestos and dried in a flat-bottomed porcelain dish to constant weight in an oven at a temperature of  $105^\circ\text{C}$ ., yields a residue of not less than 0.75 Gm. If the residue from 10 c.c. of the solution be incinerated the ash must not exceed 30 per cent. of the residue, nor must the NaCl in the ash exceed 10 per cent. of the residue when calculated from the total chlorine as determined by the U.S.P. (Volhard) method. To 100 c.c. of the solution contained in a 500 c.c. Kjeldahl flask, add 5 Gm. of  $\text{BaCO}_3$ , and 100 c.c. of water; distil 100 c.c., using a connecting bulb, into 10 c.c. of N/2HCl. Titrate the excess of acid, using cochineal as indicator, and from the acid consumed by the distillate, calculate



the percentage of N as  $\text{NH}_3$ . This must not exceed 0.35 per cent. of the total solids. Transfer 25 c.c. of the solution to a 100 c.c. Erlenmeyer flask, add 50 c.c. EtOH 94 per cent. and shake the mixture thoroughly. When the precipitate has subsided, filter, collect the precipitate upon a 9 Cm. counterpoised filter, wash it three times with a mixture of alcohol and water (2 to 1 by vol.), and then dry to constant weight at  $105^\circ\text{C}$ . The weight of this precipitate must not exceed 10 per cent. of the total solids. (Reserve the filtrate and washing for the determination of nitrogen.) To an aliquot portion of the above alcoholic filtrate from the preceding test corresponding to 1 Gm. of the alcohol soluble solids, add 4 c.c. of  $\text{H}_2\text{SO}_4$  and evaporate to dryness in a 500 c.c. Kjeldahl flask. Determine the N by the Gunning-Kjeldahl method. The amount of N thus found must not be less than 0.06 Gm.

*Faec Compressa*.—White or yellowish-white, soft and easily broken masses, having a characteristic slightly sour odour and not more than a faintly acid reaction to litmus. When examined under the microscope numerous oidium and mycoderma cells and starch grains are seen. Compressed yeast should not be used unless fresh and free from mildew and musty or foul odours.

*Ferri Hypophosphis*.—Should contain not less than 98 per cent. of  $\text{Fe}(\text{PH}_2\text{O}_2)$ . A white, or greyish-white odourless and nearly tasteless powder; permanent in the air. Soluble in 2,300 parts of water at  $25^\circ\text{C}$ ., and in 1,200 parts of boiling water; more readily soluble in the presence of  $\text{H}_3\text{PO}_3$ , or in a warm, concentrated solution of an alkali citrate, forming with the latter a green solution. When strongly heated in a dry test-tube, it evolves  $\text{PH}_3$  and leaves a residue of ferric pyrophosphate. If 10 c.c. of  $\text{HC}_2\text{H}_3\text{O}_2$  be added to 1 Gm. of the salt, no effervescence should occur, and if heated to boiling and filtered, the filtrate should respond to the following tests. A few drops of  $\text{AgNO}_3$  reagent added to a portion and warmed should give a brown to black colour or precipitate. Another portion, treated with  $\text{HgCl}_2$  solution in excess, and gently warmed, should give a white precipitate of  $\text{HgCl}$ . Another portion should give no turbidity with  $\text{Am}_2\text{C}_2\text{O}_4$  reagent. One Gm. of the salt dissolved with heat in 20 c.c. of dilute  $\text{HCl}$  should not give more than a slight turbidity with  $\text{BaCl}_2$  reagent. If 0.5 Gm. of the salt be boiled with 10 c.c. of  $\text{KOH}$  solution and the reddish-brown precipitate be removed by filtration, the filtrate slightly acidified with  $\text{HCl}$  should give no precipitate with magnesia mixture

and excess of  $\text{AmOH}$  (absence of  $\text{H}_3\text{PO}_4$ ). One Gm. of the salt is dissolved in 25 c.c. of water by the aid of sufficient  $\text{HCl}$ , added drop by drop, then 0.2 c.c. of  $\text{HNO}_3$  added and the solution boiled. On adding excess of  $\text{AmOH}$  and removing the precipitated  $\text{Fe}_2(\text{OH})_6$  by filtration, the filtrate should not respond to the U.S.P. time limit test for heavy metals. To 1 Gm. of the salt add 10 c.c. of nitro-hydrochloric acid, and evaporate to dryness. Dissolve the residue in 25 c.c. of distilled water and 15 c.c. of  $\text{HCl}$ . Transfer to a glass-stoppered container, add 4 Gm. of  $\text{KI}$  and keep at  $40^\circ\text{C}$ . for 30 minutes. Cool, and titrate with  $\text{N}/10$  hypo, with starch indicator. Each c.c. of this = 0.005584 Gm. of  $\text{Fe}$  and 0.025089 of  $\text{Fe}(\text{PH}_2\text{O}_2)_3$ . It should show not less than 22 per cent. of  $\text{Fe}$  by this test.

**Essential Oils of the B.P. 1914.** E. J. Parry. (*Chem. & Drugg.*, 1915, 86, 55.) After general criticism of the methods and processes recommended for the chemical examination of essential oils, the following are some of the specific statements made. *Oleum abietis*.—The higher limit for esters is too low. Pure oils may have sp.g. up to 0.925 and  $\alpha_D$  up to  $-43^\circ$  or even  $-45^\circ$ ; the  $\eta_D$  is too high, and this should be taken at  $20^\circ\text{C}$ . as customary and not at  $25^\circ$ . *Oleum ajowan*.—A 5 : 100 solution of  $\text{NaOH}$  for determining phenols, in this and in clove oil, must be substituted for the official 20 : 100 solution. *Oleum anisi*.—The monograph is considered to be generally unsatisfactory. *Oleum cinnamomi*.—The use of  $\text{Na}_2\text{SO}_3$  instead of  $\text{NaHSO}_3$  for determination of cinnamic aldehyde is not approved. *Oleum limonis*.—The higher limit for the  $\eta_D$  is too high; the  $\alpha_D$  would exclude half of the best oil in some seasons. *Oleum santali*.—The reduction of the  $\alpha_D$  is unnecessary since the number of pure oils with the  $\alpha_D$  below  $-16^\circ$  is so small that there should be no difficulty in the occasional bulking of such a parcel.

**Hydrogen Peroxide Solution, Determination of Acidity of, in B.P. 1914.** J. S. White. (*Pharm. J.*, 1915 [4], 40, 316.) Although the limit of acidity prescribed by the B.P. 1914 and the U.S.P. appears to be identical, similar amounts of  $\text{N}/10$   $\text{KOH}$  or  $\text{NaOH}$  being allowed to be used up, in practice the results are not identical. In the B.P. the free acid is titrated direct; in the U.S.P. excess of  $\text{N}/10$   $\text{KOH}$  is added, the  $\text{O}$  is driven off by heating, and the alkaline residue titrated back with  $\text{N}/10$   $\text{H}_2\text{SO}_4$ . The latter test is more stringent. The author

advocates the method of titration of B. L. Murray (*Y.B.*, 1914, 114), which gives accurate results.

**Ipecacuanha, Powdered, Proposed Standards for.** L. Dela ye. (*Rev. intern. pharm.*, 58, 177; *Chem. Abstr.*, 1915, 8, 2921.) To secure uniformity in pharmacopœial products, the following standards are recommended: Ash, 4-4.25; water, 10-11; EtOH extract, about 15; alkaloids, 1.9-2.10 per cent. Overstrength should be reduced by adding a sufficient amount of powdered woody portion of the root, in the place of rice powder, said to be usually employed.

**Kava, Liquid Extract of, B.P. 1914.** H. Deane. (*Pharm. J.*, 1915 [4], 40, 622.) The successive percolation of the drug, first with alcohol 90 per cent., then with alcohol 45 per cent., is strongly condemned. The extractive removed by the second percolation, with the weaker spirit, is quite insoluble in the reserved portion obtained with the stronger alcohol. The use of alcohol 45 per cent. is a waste of time, and of spirit. The whole extraction should obviously be performed, in the usual manner, with one menstruum, alcohol 90 per cent.

**Olive Oil, B.P. 1914, Test for Sesamé Oil in.** C. E. Sage. (*Pharm. J.*, 1915 [4], 50, 128.) The author considers that Baudouin's test for sesamé oil in olive oil to be unsatisfactory since perfectly genuine Spanish, Italian and North African olive oils will not respond to it. Consequently the inclusion of this test in the B.P. 1914 is criticized.

**Rhamnus Purshiana and R. Frangula, Criticism on the Chemical Test for, Proposed for the New U.S.P.** E. N. Gathercoal. (*J. Amer. Pharm. Assoc.*, 1914, 3, 982.) The "orange colour" given by a cold infusion of *R. purshiana* bark with AmOH. is also given by Alexandrian and India senna, *R. chlorophorus* bark, *R. californica* bark, commercial cascara sagrada, rumex, aloes, and rhubarb, the drugs being arranged in the order of the intensity of the reaction, with the same amount of material and reagent. The Frangula class give a red, not orange colour with this test in the following order of intensity: Commercial frangula; *R. frangula* bark; *R. crocea* bark. The test is not, therefore, distinctive of *R. purshiana*, but the difference in colour serves to distinguish *R. purshiana* from *R. frangula* bark.

**Sandalwood Oil, Solubility of, in Alcohol 70 per cent.** M. Banning and P. Van der Wielen. (*Pharm. Weekblad ; Perfum. Record*, 1915, 6, 8.) As the result of a number of critical experiments the authors conclude that the alcohol solubility test should be performed with parts by weight, and not by volume. The solubility should be determined by observing the critical point ; i.e. the temperature at which a clear solution of given weights of oil and alcohol shows turbidity when cooled. It is obvious that the strength of alcohol, when that is the solvent, must be exactly that prescribed by the test. In the case of sandalwood, it must be *exactly* 70 per cent. by volume. They consider that 1 part by weight of East Indian sandalwood should be soluble in 4 parts by weight of alcohol 70 per cent., so as to give a clear solution when cooled to 24°C. The test of the Dutch Pharmacopœia is not correct. The official test should be amended that 1 part by weight of the oil should dissolve in 5 parts by weight of 70 per cent. alcohol at 18°C. (See also *Y.B.*, 1913, 91 ; 1914, 68.)

**Scopolamine (or Hyoscine) Hydrobromide, Official Nomenclature and Characters for.** A. G. Du Mez. (*J. Amer. Pharm. Assoc.*, 1914, 86, 339.) It is suggested that the term "scopolamine hydrobromide" should be the official English title for the salt, with "hyoscine hydrobromide" as a possible synonym. As there is some doubt as to the exact m.p. of the chloroaurate of either laevo- or iso-scopolamine, this constant should not be given. Only the laevo-scopolamine hydrobromide should be officially recognized, and its specific laevorotation should be defined.

**Standards for Chemicals.** C. A. Hill. (*Chem. & Drugg.*, 1914, 85, 18.) In 1908 (*Y.B.*, 1908, 43) the author pointed out the desirability of adopting certain standards for purity and freedom from metallic contamination for a number of pharmaceutical chemicals. The majority of these have since been adopted by the authorities concerned in the compilation of the B.P. 1914. In the present paper the author tabulates the results of the analytical examination of many thousands of samples of these chemicals, showing the highest and lowest amount of the impurities found and the percentage of the chemicals which passed the standards then suggested. The figures occupy six full-page tables and represent the results of an enormous amount of work in a convenient form for reference.

**Syrups, B.P. 1914.** W. B. Cowie. (*Pharm. J.*, 1915 [4], 40, 236.) *Syrup of Ferrous Iodide*.—The addition of glucose is considered to be an advantage, but it should be directed to be mixed with the syrup, and the  $\text{FeI}_2$  solution should be filtered into the mixture. *Syrup. Ferri Phosph. c̄ Quin. et Strychnina* would be improved by the addition of 10 per cent. of glucose. *Syrup Rosae*.—The addition of 20 per cent. of EtOH to this syrup is necessary. A limit of sp.g. should be given for each syrup.

**Syrupus Hypophosphitum U.S.P., and Syrupus Hypophosphitum Co. U.S.P., New Method for Preparing.** F. A. Upsher Smith. (*Midland Drugg.*, 1915, 49, 193.) In the following formulæ the necessity for using several hypophosphites is obviated. The calcium salt is used alone, as the source of the other hypophosphites, by double decomposition.

*Syrupus Hypophosphitum Compositus, U.S.P.*—Calcium hypophosphite, 67·8 Gm.; hot distilled water, 400 c.c.; diluted sulphuric acid U.S.P., 2·2 c.c. Dissolve and add the following solution: Potassium sulphate, 14·65 Gm.; sodium sulphate, 26·58 Gm.; solution of ferric sulphate, 3·38 c.c.; manganese sulphate, 2·4 Gm.; hot distilled water, 150 c.c. When mixed add a solution of sodium citrate, 3·75 Gm.; hot distilled water, 5 c.c. Let stand over night, filter, then add quinine alkaloid, 1·1 Gm.; strychnine alkaloid, 0·115 Gm. When dissolved, add sugar, 715 Gm.; distilled water q.s. to 1,000 c.c. Dissolve sugar in cold solution, strain through cotton and add water q.s. Keep in amber bottles. A sample of the syrup made from this formula in 1913 has remained perfectly bright and clear, with an attractive yellow colour, to date.

*Syrupus Hypophosphitum U.S.P.*—Calcium hypophosphite, 70 Gm.; hot distilled water, 450 c.c. Dissolve and add diluted sulphuric acid U.S.P., 1·5 c.c. When mixed, rub in a mortar with potassium sulphate, 12·5 Gm.; sodium sulphate, dried, 10 Gm. Let stand over night, filter, and in the filtrate dissolve, in the cold, sugar, 650 Gm., previously mixed with tincture of fresh lemon peel, 5 c.c. Finally strain through cotton and add distilled water q.s. to 1,000 c.c. Keep in amber bottles.

**Tincture of Strophanthus, B.P. 1914.** H. W. Gadd. (*Pharm. J.*, 1915 [4], 39, 494.) The increase of strength in the B.P. 1914 to 4 times that of the B.P. 1898 is criticized. It is the author's experience that 60 per cent. of the batches of tincture of the

B.P. 1898 were found to be too strong when submitted to the physiological test, and had to be diluted to bring them to the B.P.C. standard. The danger is emphasized of prescribers familiar with the weaker tincture ordering this much more potent preparation in similar doses.

**Tinctures, Alcoholic Strength of, in U.S.P.** M. I. Wilbert. (*J. Amer. Pharm. Assoc.*, 1914, 3, 1660.) Alcohol 70 per cent., the strength suggested for the international standard menstruum, is considered to be much more satisfactory than the weaker alcoholic menstrua at present official in the U.S.P. Tinctures prepared with alcohol 70 per cent. retain their activity longer than those made with 50 per cent. alcohol. In the U.S. also, in many States, certain of these weaker alcoholic tinctures are merely used as "tipples." Tincture of ginger is said to be used thus. It is stated that of the 58 tinctures official in the U.S.P. not more than 25 are widely used, and of these at least 4 could be spared.

**Tolu Balsam, Pharmacopœial Test for.** (*Perfumery Record*, 1915, 6, 89.) The requirements of the new B.P. for tolu balsam are certainly stringent, and there is considerable difficulty in obtaining samples which will answer the tests prescribed. The official method of estimating the proportion of balsamic acids is not satisfactory, since the whole of the cinnamic and benzoic esters are not soluble in  $\text{CS}_2$ . By the use of the method described under "Styrax," without previous extraction with  $\text{CS}_2$ , nearly double the amount of total balsamic acids can be obtained, and the amendment of the monograph on the following lines is suggested: "2.5 Gm. of the balsam tested as described under 'Styrax Præparatus' should yield not less than 0.5 Gm. of total balsamic acids calculated as cinnamic acid."

**U.S.P. Abstracts of Proposed Changes in Standards and Descriptions in the Ninth Edition.** (*J. Amer. Pharm. Assoc.*, 1914, 3, 984, 1100, 1563.) Although the eighth edition of the *U.S.P.* has only recently been published, American pharmacists are already invited to criticize certain of the proposed changes to be published in the next edition. A few of these are reproduced here.

**GENERAL DIRECTIONS FOR ALKALOIDAL ASSAYS.**—A long series of manipulative directions is given. Methods of dealing with refractory emulsions are specially dealt with.

*Aconitum*.—No change in per cent. of alkaloids required; "aconitine" changed to "ether-soluble alkaloids of aconite." Assay as under belladonna root, using 15 Gm. of aconite in No. 40 powder and  $\text{Et}_2\text{O}$  alone as the immiscible solvent throughout the assay. Each c.c. of  $\text{N}/10 \text{ H}_2\text{SO}_4$  consumed corresponds to 64.5 Mgm. of the ether-soluble alkaloids of aconite.

*Fluid Extractum Aconiti*.—The alkaloidal yield from 100 c.c. changed from "0.4 Gm. of aconitine" to "not less than 0.45 Gm. nor more than 0.55 Gm. of the ether-soluble alkaloids." Assay: Drop from a pipette 15 c.c. of fluid extract of aconite evenly over the surface of 15 Gm. of purified sawdust (see below) and evaporate to dryness at a temperature not exceeding  $75^\circ\text{C}$ . Then transfer to a 25 c.c. flask, and proceed as directed in the assay of belladonna root, using the  $\text{AmOH}$  with a little additional water to rinse out the dish in which the mixture was evaporated, and  $\text{Et}_2\text{O}$  as the immiscible solvent throughout the assay. *Purified Sawdust*.—Moisten 1,000 Gm. of oak sawdust, in No. 20 powder, with water, pack in a cylindrical percolator and pour on enough of a  $\text{NaOH}$  1:100 to saturate the powder and leave a layer above it. When the liquid drops from the percolator, close the lower orifice and macerate the sawdust during 24 hours. Then proceed to percolate slowly until 5,000 c.c. of the sodium hydroxide solution has been added, continue the percolation with 4,000 c.c. of  $\text{HCl}$  1:100 and then wash the sawdust with water until the acid is all removed, the percolate being neutral. Finally dry the powder.

*Tinct. Aconiti*.—To contain not less than 0.045 nor more than 0.055 per cent. w/v of ether-soluble alkaloids, instead of "0.045 Gm. of aconitine from 100 c.c." The amount taken is to be 150 c.c., which is evaporated to about 20 c.c. This is incorporated with 10 Gm. of purified sawdust and dried. The assay is then conducted as described under the fluid extract.

*Extract. Aconiti Pulveratum*.—To yield not less than 1.8 nor more than 2.2 per cent. of ether-soluble alkaloids. For assay, 3 Gm. is mixed intimately in a flask with 10 Gm. of washed sand. Then add 150 c.c. of  $\text{Et}_2\text{O}$  and 2 c.c. of  $\text{AmOH}$ ; shake vigorously every 10 minutes, during half an hour; allow to subside; decant 100 c.c. and treat as described under the fluid extract.

*Belladonna Folia*.—Only slight modifications suggested.

*Tinct. Belladon. Fol.*—To contain not less than 0.027 nor

more than 0.033 per cent. w/v. For assay, 100 c.c. evaporated to 10 c.c. is treated as described under belladonna root, with slight modification.

*Extract. Belladon. Fol.*—To yield not less than 1.18 nor more than 1.32 per cent. of mydriatic alkaloids. Two Gm. to be taken for assay, dissolved in 10 c.c. of dilute alcohol (48.6 per cent.) transferred to a separator, another 10 c.c. in portions being used to wash. Then proceed as *Ext. Belladon. Fluid*.

*Extract. Belladon. Fol. Pulv.*—To yield not less than 1.18 nor more than 1.32 per cent. of mydriatic alkaloids. Three Gm. taken for assay mixed with 10 Gm. of pure sand. Then treated with 150 c.c. of a mixture of  $\text{CHCl}_3$ , 1 vol., with  $\text{Et}_2\text{O}$ , 2 vols.; add 2 c.c. of AmOH 10 per cent. Agitate frequently for 30 minutes, then decant 100 c.c. and proceed as described under the root, modifying the process there given by treating the residue twice with  $\text{Et}_2\text{O}$ , before titrating, evaporating to dryness each time.

*Belladonnæ Radix.—Assay:* Introduce 15 Gm. of belladonna root, in No. 60 powder, into a flask of about 300 c.c. capacity and add 150 c.c. of a mixture of  $\text{CHCl}_3$ , 1 vol., and  $\text{Et}_2\text{O}$ , 2 vols. Stopper the flask, shake it well and allow it to stand 10 minutes, then add 5 c.c. of AmOH 10 per cent. and shake the flask vigorously every 10 minutes during 2 hours. Now add 15 c.c. of distilled water, again shake the flask well, and, when the drug has settled, decant 100 c.c. of the solution, representing 10 Gm. of belladonna root. Filter the solution through a pledget of purified cotton into a separator, and rinse the graduate and cotton with a little  $\text{Et}_2\text{O}$ . Completely extract the alkaloids from the  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  solution by shaking out repeatedly with dilute  $\text{H}_2\text{SO}_4$ . Collect the acid washings in a separator, add AmOH 10 per cent. until the solution is decidedly alkaline to litmus, and completely extract the alkaloids by shaking out repeatedly with  $\text{CHCl}_3$ . Evaporate the combined  $\text{CHCl}_3$  washings to dryness and dissolve the alkaloids from the residue in exactly 5 c.c. of N/10  $\text{H}_2\text{SO}_4$ , and titrate the excess of acid with N/50 KOH, using cochineal as indicator. Each c.c. of N/10  $\text{H}_2\text{SO}_4$  consumed = 28.92 Mgm. of the mydriatic alkaloids.

*Fluid Extractum Belladonnæ Radicis.*—The alkaloidal yield from 100 c.c. to be not less than 0.405 Gm. nor more than 0.495 Gm. of the mydriatic alkaloids. *Assay:* Introduce 10 c.c. into a separator and add 10 c.c. of water and



2 c.c. of AmOH 10 per cent. Completely extract the alkaloids by shaking out repeatedly with  $\text{CHCl}_3$  and then extract the alkaloids from the  $\text{CHCl}_3$  solution by shaking out repeatedly with dilute  $\text{H}_2\text{SO}_4$ . Collect the acid washings in a separator, render them alkaline with AmOH, and shake out with successive washings of  $\text{CHCl}_3$ . Then proceed as described under belladonna root.

*Cantharis*.—It should yield not less than 0.6 per cent. of cantharidin. *Assay*: Introduce 15 Gm. of cantharides, in No. 40 powder, into a stout bottle of not less than 250 c.c. capacity, add 150 c.c. of a mixture of  $\text{C}_6\text{H}_6$ , two vols., and petroleum ether, 1 vol., and then add 2 c.c. of HCl. Stopper the bottle tightly, shake it well, and allow it to stand over night. Now gradually warm the bottle and its contents to about  $40^\circ\text{C}$ . and maintain it at that temperature with frequent shaking for 3 hours. Cool, filter off 100 c.c. of clear solution and evaporate rapidly to about 5 c.c. Now add 5 c.c. of  $\text{CHCl}_3$  and set aside in a moderately warm place. When the solvent has all evaporated, add to the crystals 10 c.c. of a mixture of equal volumes of absolute EtOH and purified petroleum benzin, which has previously been saturated with pure cantharidin, allow it to stand during 15 minutes and then decant it through a pellet of purified cotton. Wash the crystals with successive portions of a saturated solution of cantharidin, similar to that directed above, until it is free from fat and colouring matter, and pass the washings through the same pellet of purified cotton. Then wash the cotton with a very small quantity of warm  $\text{CHCl}_3$  to dissolve any adhering crystals, collect the  $\text{CHCl}_3$  in the vessel containing the washed crystals, evaporate off the solution with the aid of a blast of air, dry at  $60^\circ\text{C}$ . for half an hour and weigh. The result, the cantharidin in 10 Gm. of cantharides.

*Cinchona*.—To contain not less than 6 per cent. of the total alkaloids. *Assay*: Introduce 5 Gm. of bark, in No. 40 powder, into a 500 c.c. flask and add 200 c.c. of a mixture of  $\text{CHCl}_3$ , 1 vol., and  $\text{Et}_2\text{O}$ , 2 vols. Stopper the flask, shake it well, and let it stand 10 minutes. Then add 5 c.c. of AmOH, shake the flask frequently for 1 hour, and let it stand over night. Now add 10 c.c. of distilled water, shake the mixture vigorously, and when the drug has settled decant 160 c.c. of the solution, representing 4 Gm. of cinchona. Filter it through a pledget of purified cotton into a separator, and rinse both cylinder and

cotton with  $\text{Et}_2\text{O}$ . Completely extract the alkaloids from the  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  solution by shaking out repeatedly with weak  $\text{H}_2\text{SO}_4$ . Collect the acid solutions in a separator, add  $\text{AmOH}$  until the solution is distinctly alkaline to litmus, and completely extract the alkaloids by shaking out repeatedly with  $\text{CHCl}_3$ . Filter each portion of  $\text{CHCl}_3$  as it comes from the separator through a pledget of purified cotton into a tared flask, and wash the funnel and cotton with  $\text{CHCl}_3$ . Evaporate off the  $\text{CHCl}_3$ , add 5 c.c. of  $\text{EtOH}$  to the residue, and again evaporate. Repeat the evaporation with  $\text{EtOH}$  and dry the residue at  $100^\circ\text{C}$ . to constant weight. The weight will be the amount of total alkaloids from 4 Gm. of bark.

*Fluid Extractum Cinchonæ*.—To contain not less than 4.5 nor more than 5.5 per cent. w/v of total alkaloids. *Assay*: Drop from a pipette 5 c.c. of fluid extract of cinchona evenly over the surface of 10 Gm. of purified sawdust and evaporate it to dryness at a temperature not exceeding  $80^\circ\text{C}$ . Then transfer the mixture to a 500 c.c. flask, and proceed as directed in the assay of cinchona, modifying the process there given by increasing the  $\text{AmOH}$ , 10 per cent., to 10 c.c. Use this in divided portions to rinse the dish in which the mixture was evaporated and add the rinsings to the flask.

*Tinct. Cinchonæ*.—To contain not less than 0.9 nor more than 1.1 per cent. w/v of total alkaloids. *Assay*: Evaporate 25 c.c. on the water-bath until it measures 15 c.c. Incorporate 10 Gm. of purified sawdust and dry at  $80^\circ\text{C}$ . Treat the dried sawdust as directed under cinchona, increasing the amount of  $\text{AmOH}$  to 10 c.c., using this to wash out the evaporation dish.

*Cinchona Rubra*.—To contain not less than 6 per cent. of total alkaloids.

*Tinct. Cinchonæ Co.*—To contain not less than 0.45 nor more than 0.55 per cent. w/v of total alkaloids. Take 50 c.c. of tincture for assay, then proceed as under *tinct. cinchonæ*.

*Colchici Cormus*.—Fifteen Gm. taken for assay as under *colchicum seed*.

*Extractum Colchici Pulverat.*—To yield not less than 1.25 nor more than 1.55 per cent. of colchicine. For assay take 6 Gm. of the powdered extract and proceed as under *colchicum seed*.

*Colchici Semen*.—*Assay*: Introduce 15 Gm. of the drug, in No. 60 powder, into a 500 c.c. flask; add 300 c.c. of a mixture

of 10 c.c. of basic lead acetate solution and 290 c.c. of distilled water. Weigh the flask and contents and digest at  $60^{\circ}$ – $70^{\circ}\text{C}$ . for 3 hours with occasional agitation; cool, make up to weight and filter off 200 c.c. Add 0.75 Gm. of  $\text{Na}_2\text{HPO}_4$ , shake repeatedly for 30 minutes, then filter off 100 c.c. (=5 Gm. of drug). Shake out with  $\text{CHCl}_3$  in successive lots until the  $\text{CHCl}_3$  gives no precipitate with I. The bulked  $\text{CHCl}_3$  is evaporated in a tared flask, the residue being treated with three successive 1 c.c. of EtOH, and evaporated off separately. The residue is then dried to constant weight at  $100^{\circ}\text{C}$ . After weighing it is treated with 5 c.c. of N/10  $\text{H}_2\text{SO}_4$  and 5 c.c. of water, and heated to  $70^{\circ}\text{C}$ . The solution is filtered through a plug of cotton, which is then washed with water, the aqueous liquid being rejected. Any insoluble residue transferred to the wool is washed back into the flask by treating it first with EtOH and then with  $\text{Et}_2\text{O}$ . The solvent is evaporated and the dry residue weighed. The difference between the two weighings is taken as colchicine.

*Fluid Extractum Colchici Seminis*.—To yield not less than 0.036 nor more than 0.044 per cent. w/v of colchicine. *Assay*: Introduce 15 c.c. into a 500 c.c. flask, add 10 c.c. of solution of lead subacetate, previously diluted with 35 c.c. of water, shake the mixture thoroughly, then add 240 c.c. of water, again agitate the mixture and proceed as directed in the assay of colchicum seed.

*Tinctura Colchici Seminis*.—To yield not less than 0.036 nor more than 0.044 per cent. w/v of colchicine. *Assay*: Evaporate 150 c.c. on a water-bath to about 20 c.c., transfer it to a 50 c.c. graduated flask and rinse the evaporating dish with about 10 c.c. of water in divided portions. Then add 10 c.c. of solution of lead subacetate, shake the mixture thoroughly, add enough distilled water to make 50 c.c. and pour this into a 500 c.c. flask. Now add 250 c.c. of recently boiled distilled water, using part of the water to rinse the flask and proceed as directed in the assay of colchicum seed.

*Guarana*.—To yield not less than 4 per cent. of caffeine. *Assay*: Introduce 6 Gm., in No. 60 powder, into a flask and add 120 c.c. of  $\text{CHCl}_3$  and 6 c.c. of AmOH 10 per cent. Stopper the flask, shake it frequently for half an hour, then let it stand 4 hours. Again shake the flask vigorously and when the drug has settled, filter the liquid rapidly through a pledget of purified cotton and collect 100 c.c. of the filtrate, representing 5 Gm. of drug. Evaporate the clear filtrate to dryness and

dissolve the residue in dilute  $\text{H}_2\text{SO}_4$  with the aid of heat. When the liquid has cooled, filter it into a separator and wash the container and filter with several small portions of distilled water. Now add  $\text{AmOH}$ , 10 per cent., until the liquid is distinctly alkaline to litmus and shake out with  $\text{CHCl}_3$  until completely extracted, as shown by testing with I reagent. Evaporate the united  $\text{CHCl}_3$  solutions and dry the residue to constant weight at  $100^\circ\text{C}$ . Weigh as total caffeine from 5 Gm. of drug.

*Fluid Extractum Guaranae*.—To yield not less than 3.6 nor more than 4.4 per cent. w/v of caffeine. *Assay*: Introduce 5 c.c. into a separator, add 1 c.c. of  $\text{AmOH}$  10 per cent., and shake out the alkaloid with  $\text{CHCl}_3$  until completely extracted, as shown by testing with I reagent. Evaporate the combined  $\text{CHCl}_3$  solutions to dryness and dissolve the residue in 20 c.c. of distilled water with the aid of heat. Allow this to cool, filter it into a separator and wash the container and filter with several small portions of distilled water, adding the rinsings to the liquid in the separator. Then shake out the alkaloid with  $\text{CHCl}_3$  until completely extracted, as shown by testing with I reagent, evaporate the combined  $\text{CHCl}_3$  solutions, dry at  $100^\circ\text{C}$  and weigh.

*Hydrastis*.—Assay as under belladonna root, taking 10 Gm. of drug in No. 60 powder, and 100 c.c. of  $\text{Et}_2\text{O}$ ; withdrawing 50 c.c. of the  $\text{Et}_2\text{O}$  solution (=5 Gm. of drug). Use  $\text{Et}_2\text{O}$  alone as the immiscible solvent throughout the assay, and dry the residue to constancy at  $100^\circ\text{C}$ . Express result as  $\text{Et}_2\text{O}$ -soluble alkaloids, not as hydrastine.

*Fluid Extractum Hydrastis*.—To yield not less than 1.8 nor more than 2.2 per cent. w/v. For assay, take 5 c.c. and proceed as in fluid extract of belladonna, but use  $\text{Et}_2\text{O}$  alone throughout the process. Dry residue at  $100^\circ\text{C}$ . and weigh.

*Glyceritum Hydrastis*.—To yield not less than 1.12 nor more than 1.37 per cent. w/v of  $\text{Et}_2\text{O}$  soluble alkaloids. Proceed with 5 Gm. of material as in fluid extract of hydrastis, but wash the final  $\text{Et}_2\text{O}$  extractions with 10 c.c. of water and discard the aqueous portion. Filter the  $\text{Et}_2\text{O}$  portion through cotton.

*Tinct. Hydrastis*.—To yield not less than 0.36 nor more than 0.44 per cent. w/v of  $\text{Et}_2\text{O}$ -soluble alkaloids. Evaporate 50 c.c. to 10 c.c.; transfer to a separator and proceed as in fluid extract of belladonna root, but using the  $\text{AmOH}$ , 10 per cent., diluted with 5 c.c. of water, to wash out the evaporation capsule. Use  $\text{Et}_2\text{O}$  alone throughout; dry the  $\text{Et}_2\text{O}$  residue at  $100^\circ\text{C}$ . and weigh.

*Extractum Hydrastis Pulveratum.*—To yield not less than 9 nor more than 11 per cent. of  $\text{Et}_2\text{O}$ -soluble alkaloids. Take 3 Gm. for assay, add 10 Gm. of pure sand to it in a flask; then add 150 c.c. of  $\text{Et}_2\text{O}$  and 5 c.c. of  $\text{AmOH}$  10 per cent. Agitate every 10 minutes for half an hour; allow to subside and withdraw 100 c.c. of the clear  $\text{Et}_2\text{O}$  solution (=2 Gm. of extract). Proceed as in the assay of belladonna root, but using  $\text{Et}_2\text{O}$  alone throughout the assay, and drying the residue to constant weight at  $100^\circ\text{C}$ . The weight= $\text{Et}_2\text{O}$ -soluble alkaloids from 2 Gm. of extract.

*Hyoscyamus.*—To yield 0.06 per cent. of mydriatic alkaloids. Assay as under belladonna root, using 30 Gm. of drug, in No. 60 powder, and 300 c.c. of the  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  mixture. Increase the amount of distilled water, added after maceration, to 40 c.c. and take 200 c.c. of the  $\text{CHCl}_3$ - $\text{Et}_2\text{O}$  solution=20 Gm. of drug. Before titration, treat the final residue twice with 5 c.c. of  $\text{Et}_2\text{O}$ , evaporating to dryness each time.

*Fluid Extractum Hyoscyami.*—To yield not less than 0.055 nor more than 0.075 per cent. w/v of mydriatic alkaloids. Proceed as in the fluid extract of belladonna root, using 25 c.c. of the fluid extract; before titrating treat the residue twice with 5 c.c. of  $\text{Et}_2\text{O}$ , evaporating to dryness each time.

*Tinctura Hyoscyami.*—To yield not less than 0.0055 and not more than 0.0075 per cent. w/v of total mydriatic alkaloids. Assay: Evaporate 250 c.c. on a water-bath to about 10 c.c., transfer to a separator and proceed as directed under fluid extract of belladonna root, but increasing the  $\text{AmOH}$  to 5 c.c., which, with 5 c.c. of water, is to be used, in divided portions, to wash the evaporation capsule; before titrating treat the residue twice with 5 c.c. of  $\text{Et}_2\text{O}$  evaporating to dryness each time.

*Extractum Hyoscyami.*—To yield not less than 0.215 nor more than 0.288 per cent. of mydriatic alkaloids. Assay: Proceed as directed with belladonna leaves, taking 5 Gm. of extract for the assay.

*Ipecacuanha.*—To yield 2 per cent. of alkaloids. Assay as under belladonna root, using 10 Gm. of drug, in No. 80 powder, and 100 c.c. of  $\text{Et}_2\text{O}$ . Take 50 c.c. of the  $\text{Et}_2\text{O}$  solution, representing 5 Gm. of ipecac. Use  $\text{Et}_2\text{O}$  alone as the immiscible solvent throughout and dissolve the residue in 10 c.c. of  $\text{N}/10 \text{ H}_2\text{SO}_4$ . Each c.c. of  $\text{N}/10 \text{ H}_2\text{SO}_4$ =0.024 Gm. of alkaloids.

*Fluid Extractum Ipecacuanhæ.*—To yield not less than 1·8 nor more than 2·2 per cent. w/v of the  $\text{Et}_2\text{O}$ -soluble alkaloids. Drop from a pipette 10 c.c. of fluid extract of ipecac. evenly over the surface of 10 Gm. of purified sawdust, and evaporate it to dryness at a temperature not exceeding  $80^\circ\text{C}$ . Then transfer the mixture to a 250 c.c. flask, and proceed as under fluid extract of belladonna. But increase the AmOH 10 per cent. to 5 c.c. and use it diluted with 5 c.c. of water to wash out the evaporation capsule. Then add 100 c.c. of  $\text{Et}_2\text{O}$ , and take 50 c.c. of the  $\text{Et}_2\text{O}$  solution (= 5 c.c. of liquid extract). Use  $\text{Et}_2\text{O}$  alone throughout. Dissolve the residue in N/10  $\text{H}_2\text{SO}_4$ , and titrate back with N/50 KOH. Each 1 c.c. of N/10  $\text{H}_2\text{SO}_4$  combined = 0·024 Gm. of ipecacuanha alkaloids.

*Jalap.*—The requirement that not more than 15 per cent. of the total resin should be soluble in  $\text{Et}_2\text{O}$  is omitted. *Assay* : Percolate 10 Gm. of the drug in No. 60 powder with EtOH 94 per cent. to obtain 100 c.c. of percolate. Transfer 20 c.c. of this to a separator, add 20 c.c. of  $\text{CHCl}_3$ , mix, add 20 c.c. of water and shake thoroughly. After separation, draw off the  $\text{CHCl}_3$  into a tared beaker. Evaporate on the water-bath ; to the residue add 2 c.c. of EtOH and again evaporate. Dry to constancy at  $100^\circ\text{C}$ . The weight = the resin in 2 Gm. of jalap.

*Nux Vomica.*—To yield not less than 2·5 per cent. of total alkaloids. Assay as under belladonna root, taking 15 Gm. of drug, and increasing the AmOH to 10 c.c. and the time of maceration to 12 hours. For titration of the final residue, dissolve in 10 c.c. of N/10  $\text{H}_2\text{SO}_4$ . Each c.c. of N/10  $\text{H}_2\text{SO}_4$  combined = 0·0364 Gm. of nux vomica alkaloids.

*Fluid Extract. Nucis Vom.*—To yield not less than 2·37 nor more than 2·63 per cent. w/v of total alkaloids. Proceed as under fluid extract of belladonna, but use 10 c.c. of N/10  $\text{H}_2\text{SO}_4$  to dissolve the final residue for titration.

*Tinct. Nucis Vom.*—To yield not less than 0·237 nor more than 0·263 per cent. w/v. For assay, evaporate 100 c.c. on the water-bath to 10 c.c. Transfer to a separator, then proceed as in fluid extract of belladonna root, but use 5 c.c. of AmOH 10 per cent. diluted with water, to wash out the evaporation capsule. Dissolve the final residue in 10 c.c. of N/10  $\text{H}_2\text{SO}_4$  for titration.

*Extractum Nucis Vomice Pulveratum.*—The alkaloidal yield changed from " 5 per cent. of strychnine " to " not less than 15·2 per cent. nor more than 16·8 per cent. of the total alkaloids

of nux vomica." Introduce 3 Gm. of powdered extract of nux vomica into a 250 c.c. flask, add 10 Gm. of washed sand and mix thoroughly. Then add 150 c.c. of a mixture of  $\text{Et}_2\text{O}$ , 2 vols., and  $\text{CHCl}_3$ , 1 vol., and 5 c.c. of  $\text{AmOH}$ . Shake the mixture vigorously every 10 minutes during half an hour, and when the dregs have settled decant 100 c.c. of the clear liquid, representing 2 Gm. of the extract. Proceed as directed in the assay of belladonna root, modifying the process there given by dissolving the alkaloidal residue in 10 c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4$  instead of 5 c.c.

*Physostigma*.—No change in alkaloidal standard. *Assay*: Introduce 15 Gm. of physostigma in No. 60 powder, into a flask of about 300 c.c. capacity, and add 150 c.c. of  $\text{Et}_2\text{O}$ . Stopper the flask, shake it well and allow it to stand 10 minutes, then add 10 c.c. of an aqueous solution of  $\text{NaHCO}_3$  (1 in 20) and shake the mixture vigorously at intervals during 4 hours. Now add 15 c.c. of distilled water, again shake the flask well, and when the drug has settled, decant 100 c.c. of the solution, representing 10 Gm. of physostigma. Filter the solution through a pledget of purified cotton into a beaker, and rinse the graduate and cotton with  $\text{Et}_2\text{O}$ . Add 20 c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4$  and evaporate off the  $\text{Et}_2\text{O}$ , stirring during the evaporation with a rubber-tipped glass rod. After the resinous and fatty matter has agglutinated, pour off the acid solution through a wetted filter into a separator. Redissolve the residue in the beaker in about 15 c.c. of  $\text{Et}_2\text{O}$ , add 2 c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4$ , evaporate off the  $\text{Et}_2\text{O}$  with continued stirring as before and pour the acid solution on the filter. Repeat this operation until all of the alkaloid is extracted and then wash the filter with distilled water until it is free from alkaloids. Collect the solution and washings in a separator, add sufficient  $\text{NaHCO}_3$  to make the solution decidedly alkaline to litmus and completely extract the alkaloids by shaking it out repeatedly with  $\text{Et}_2\text{O}$ . Wash the combined  $\text{Et}_2\text{O}$  with 10 c.c. of distilled water and filter the  $\text{Et}_2\text{O}$  solution, washing the container and filter with more  $\text{Et}_2\text{O}$ . Evaporate the combined  $\text{Et}_2\text{O}$  solutions to dryness, dissolve the alkaloids from the residue in exactly 5 c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4$  and titrate the excess of acid with  $\text{N}/50 \text{KOH}$ , using cochineal reagent as indicator. Each c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4$  consumed corresponds to 0.02752 Gm. of the alkaloids of physostigma.

*Tinctura Physostigmatidis*.—The alkaloidal yield from 100 c.c.

changed from "0.014 Gm. of the  $\text{Et}_2\text{O}$ -soluble alkaloids from physostigma" to "not less than 0.013 Gm. nor more than 0.017 Gm. of the alkaloids of physostigma." Evaporate 150 c.c. of tincture of physostigma on a water-bath until it measures about 20 c.c., add 10 Gm. of purified sawdust to the liquid and incorporate it thoroughly. Continue the evaporation on a water-bath, until the mixture is dry, then transfer the impregnated sawdust to a 250 c.c. flask, and proceed as directed in the assay of physostigma, modifying the process there given by using the solution of  $\text{NaHCO}_3$ , in divided portions, to rinse the evaporating dish.

*Extractum Physostigmatis*.—To yield not less than 1.7 nor more than 2.3 per cent. of total alkaloids. Take 2 Gm. for assay, dissolve in 10 c.c. of dilute  $\text{EtOH}$ ; drop the solution evenly over 15 Gm. of purified sawdust, wash out the vessel with another 5 c.c. of dilute  $\text{EtOH}$  and add this to the sawdust; evaporate to dryness on the water bath, transfer to a 250 c.c. flask and proceed as directed under physostigma.

*Pilocarpus*.—To yield 0.6 per cent. of total alkaloids. For assay take 15 Gm. of pilocarpus in No. 60 powder and proceed as directed under belladonna root, decreasing the amount of water added after maceration to 5 c.c. Each c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4 = 0.0208$  Gm. of pilocarpus alkaloids.

*Fluid Extractum Pilocarpi*.—To yield not less than 0.55 nor more than 0.65 per cent. w/v of total alkaloids. Fifteen c.c. taken and dropped on 15 Gm. of purified sawdust, dried, and treated as directed under belladonna root, increasing the  $\text{AmOH}$  10 per cent. to 5 c.c. and using it and 5 c.c. of water to wash evaporating capsule.

*Stramonium*.—Assay as directed for belladonna root, taking 15 Gm. of drug in No. 50 powder, and evaporating the final residue twice with 5 c.c. of  $\text{Et}_2\text{O}$  before titration. Each c.c. of  $\text{N}/10 \text{H}_2\text{SO}_4 = 0.02892$  Gm. of mydriatic alkaloids.

*Tinct. Stramonii*.—To yield not less than 0.0225 nor more than 0.0275 per cent. w/v of mydriatic alkaloids. For assay, evaporate 100 c.c. to 10 c.c. on water-bath; transfer to separator; proceed as under fluid extract of belladonna, but using 5 c.c. of water to wash evaporating capsule and evaporating the final residue twice with  $\text{Et}_2\text{O}$  before titrating.

*Extractum Stramonii*.—To yield not less than 0.9 nor more than 1.1 per cent. of total mydriatic alkaloids. Take 2 Gm. for assay and proceed as with extract of belladonna. .



*Extract. Stramonii Pulveratum.*—To yield not less than 0.9 nor more than 1.1 per cent. of total alkaloids. Take 3 Gm. for assay and proceed as with powdered extract of belladonna.

### BIOLOGICAL PRODUCTS.

Modifications to the monograph on *Serum antidiphthericum* are suggested, the addition "sometimes shows a slight granular sediment" is put forward. Also that only such serums as have been prepared and standardized in officially licensed laboratories may be sold, and the strongest regulations as to the label bearing the date, name of laboratory, etc., are to be followed. Other monographs for sera suggested for inclusion are *Serum antidiphthericum purificatum*; *Serum antidiphthericum siccum*; *Serum antitetanicum*; *Serum antitetanicum purificatum*, and *siccum*; and *Virus vaccenicum*. All these are to be kept in amber-coloured glass containers free from air, in a dark place, at a temperature between 4.5° and 15°C.

*Glandulæ Suprarenales Siccæ.*—The suprarenal glands of such animals as are used for food by man, cleaned, dried, freed from fat, and powdered, and yielding not less than 0.4 per cent. nor more than 0.6 per cent. of epinephrine. To contain not more than 7 per cent. of moisture. *Assay*: Add 0.005 Gm. of finely powdered  $MnO_2$  and 10 c.c. of distilled water to 0.010 Gm. of desiccated suprarenal glands; thoroughly shake the mixture several times during an hour and filter. Compare the colour of the filtrate in a test-tube or in any convenient manner, with the colour of standard solutions made as follows: Mix 1.85 c.c. of  $CoCl_2$  reagent with 0.95 c.c. of  $AuCl_3$  reagent and 7.2 c.c. of distilled water; the colour corresponds to 0.2 per cent. of epinephrine in the filtrate obtained above; 2.95 c.c. of  $CoCl_2$  reagent with 1.25 c.c. of  $AuCl_3$  reagent and 5.8 c.c. of distilled water corresponds in colour to 0.4 per cent. of epinephrine; 4.05 c.c. of  $CoCl_2$  reagent with 1.35 c.c. of  $AuCl_3$  reagent and 4.6 c.c. of distilled water corresponds in colour to 0.6 per cent. of epinephrine; 5.15 c.c. of  $CoCl_2$  reagent with 1.55 c.c. of  $AuCl_3$  reagent and 3 c.c. of distilled water corresponds in colour to 0.8 per cent. of epinephrine. The percentages of epinephrine indicated by the above colour standards are based upon the maceration of 0.010 Gm. of the desiccated suprarenal glands in 10 c.c. of distilled water as directed above and filtering. In samples containing more

than 0·8 per cent. of epinephrine, 0·005 Gm. of the desiccated suprarenal glands may be taken, in which case the percentage stated above, as indicated by the colour standards, should be doubled. The standard colour solutions keep unchanged indefinitely if sealed in test-tubes. The former test with  $\text{Fe}_2\text{Cl}_6$  is to be omitted. The test solutions required above are made as follows: *Cobaltous Chloride T.S.*—Two Gm. of  $\text{CoCl}_2$  dissolved with the aid of 1 c.c. of  $\text{HCl}$  in sufficient distilled water to measure 100 c.c. *Gold Chloride T.S.*—An aqueous solution of  $\text{AuCl}_3$  containing 0·1 Gm. of  $\text{Au}$  in each 100 c.c. of solution, determined by analysis.

*Glandulæ Thyroidæ Siccæ.*—The thyroid glands of such animals as are used for food by man, freed from connective tissue and fat, dried and powdered and yielding not less than 0·17 per cent. nor more than 0·23 per cent. of  $\text{I}$  in thyroid combination. Iodine in inorganic or any other form of combination than that peculiar to the thyroid must be absent. Not to contain more than 6 per cent. of moisture. Ash, 5 per cent. *Assay*: Mix 1 Gm. of desiccated thyroid glands in a nickel crucible of about 125 c.c. capacity, with 15 Gm. of a mixture composed of 138 parts by weight of anhydrous  $\text{K}_2\text{CO}_3$ , 106 parts of anhydrous  $\text{Na}_2\text{CO}_3$  and 75 parts of  $\text{KNO}_3$ . Spread an additional 5 Gm. of this fusion mixture evenly over the surface. Heat the crucible over a Bunsen flame until no further carbonization is observed, cool it and dissolve the residue in about 150 c.c. of distilled water, warming to hasten solution. Transfer this solution to a 500 c.c. Erlenmeyer flask, and add approximately 50 c.c. of a fresh solution of chlorinated soda. Now treat the mixture with enough  $\text{H}_3\text{PO}_4$ , diluted with an equal volume of distilled water, to produce an appreciable yellow tint of free chlorine, then add 10 c.c. more of the  $\text{H}_3\text{PO}_4$ , diluted with an equal volume of distilled water and boil the contents of the flask for half an hour or until the volume has been reduced to about 150 c.c. Cool the liquid, add 10 c.c. of an aqueous solution of  $\text{KI}$  (1 in 100) and titrate the liberated iodine with  $\text{N}/100$  hypo with starch indicator added just before the end of the titration.

#### ESSENTIAL OILS.

The following alterations are suggested:—

*Methylis Salicylas.*—To yield not less than 98 per cent. of methyl salicylate ( $\text{CH}_3\text{C}_7\text{H}_5\text{O}_2=152\cdot06$ ). The synthetic pro-

duct ; or that obtained by distillation from *Betula lenta* or from *Gaultheria procumbens*. The source from which it is derived, in every case must be stated on the label. A colourless, yellowish or reddish liquid, having the characteristic odour and taste of gaultheria. Sp.g. at 25°C. ; synthetic, 1.180 to 1.185 ; when from sweet birch or gaultheria, 1.172 to 1.180 ; b.p. 218° to 221°C. Synthetic methyl salicylate, or that from sweet birch, is optically inactive ; when obtained from gaultheria, it is slightly laevogyrate, not exceeding  $-1.5^\circ$  at 25°C. Soluble in 6 volumes of 70 per cent. alcohol at 25°C. with not more than a slight cloudiness. The alcoholic solution is neutral or slightly acid to moistened litmus paper. Add 10 c.c. of KOH reagent to 1 c.c. of methyl salicylate, contained in a capacious test-tube, and agitate the mixture. A clear colourless or faintly yellowish solution results, without the separation of any oily drops either on the surface or at the bottom of the liquid (other volatile oils or petroleum). It does not respond to the volatile oil heavy metals test. *Assay* : for methyl salicylate : Introduce about 2 c.c. of methyl salicylate into a tared flask, note the exact weight, add 50 c.c. of alcoholic N/2 KOH, connect the flask with a reflux condenser and heat the mixture on a water-bath during 2 hours. Then add a few drops of phenolphthalein, and titrate the excess of alkali with N/2 HCl. *Volatile Oil Heavy Metal Test*.—Shake 10 c.c. of the oil with an equal volume of distilled water and pass  $H_2S$  to saturation through the liquid. No darkening in colour should be produced either in the oil or in the water.

*Oleum Amygdalæ Amaræ*.—Obtained by maceration and distillation from the ripe kernels of *Prunus amygdalus* var. *amara*, and from other kernels containing amygdalin, the source from which it is derived in every case to be stated on the label ; yielding not less than 85 per cent. of benzaldehyde and not less than 2 per cent. nor more than 4 per cent. of HCN. This oil is intended for medicinal use ; it is not to be used for flavouring foods. Sp.g. from 1.038 to 1.060 at 25°C. ;  $\eta_D$  1.5428 to 1.5439 at 20°C. Optically inactive or dextrogyrate, not exceeding  $+0^\circ 10'$  at 25°C. Dissolves, forming a clear solution, in 2 volumes of 70 per cent. EtOH. Add 10 drops of the oil to a little EtOH, introduce a small amount of zinc dust and 2 c.c. of  $HC_2H_3O_2$  and boil the mixture for a short time ; no odour of phenyl isonitrile should develop after rendering it strongly alkaline with KOH, adding a drop of  $CHCl_3$ .

and heating (nitro-benzene). *Assay for benzaldehyde*: Dissolve 3 Gm. of phenylhydrazine (not darker in colour than pale yellow) in 50 c.c. of EtOH and titrate 25 c.c. of the solution with N/2 HCl, using methyl orange indicator, to a distinct change in colour. To about 1 Gm. of oil of bitter almond, accurately weighed, add 25 c.c. of the phenylhydrazine solution just prepared and titrate the mixture after 30 minutes with N/2 HCl as just described. The difference between the number of c.c. required in the two titrations,  $\times 0.053$  will show the weight of benzaldehyde present. Always freshly prepare the phenylhydrazine solution when required for the assay. *Assay for hydrocyanic acid*: Dissolve 15 Gm. of  $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$  in enough distilled water to measure 100 c.c., add 5 c.c. of this solution to 40 c.c. of distilled water, 5 c.c. of N/2 NaOH and 2 drops of  $\text{K}_2\text{CrO}_4$  solution and titrate the solution with N/10  $\text{AgNO}_3$ , to the production of a permanent reddish tint. Pour this mixture into a 100 c.c. flask containing about 1 Gm. of oil of bitter almond, accurately weighed, and titrate again with N/10  $\text{AgNO}_3$  until a red tint, which does not disappear on shaking, is reproduced.

*Oleum Anisi*.—Sp.g. from 0.978 to 0.988 at  $25^\circ\text{C}$ .;  $\eta_{\text{D}20}$  1.5440 to 1.5600.  $\alpha_{\text{D}}$  varies from  $+1^\circ$  to  $-2^\circ$  at  $25^\circ\text{C}$ . (oil of fennel). Soluble with not more than a slight cloudiness in 3 volumes of EtOH 90 per cent. Cool the oil in determining congealing point to  $12^\circ\text{C}$ . It does not respond to the volatile oil heavy metals test.

*Oleum Aurantii Corticis*.—The  $\eta_{\text{D}}$  1.4723 to 1.4737 at  $20^\circ\text{C}$ .;  $\alpha_{\text{D}}$  not less than  $+95^\circ$ . Fractionation test thus specified: Introduce 50 c.c. into a 200 c.c. 3-bulb Ladenburg flask of approximately the following dimensions: The lower or main bulb, 6 cm. in diameter; the smaller or condensing bulbs, 3.5 cm., 3 cm. and 2.5 cm. in diameter, with the distance of 20 cm. from the bottom of the flask to the side arm. Distil the oil at the rate of one drop a second until 5 c.c. has been obtained. The  $\alpha_{\text{D}}$  of the first 10 per cent. thus obtained is equal to or slightly greater than that of the original sample and the  $\eta_{\text{D}}$  not less than 0.0008 nor more than 0.0015 lower than that of the original, at  $20^\circ\text{C}$ . The nitroso-limonene test for added turpentine is omitted.

*Oleum Cadinum*.—Sp.g. 0.980 to 1.055 at  $25^\circ\text{C}$ . Completely soluble in 3 vols. of  $\text{Et}_2\text{O}$ ,  $\text{C}_5\text{H}_{11}\text{OH}$ ,  $\text{CHCl}_3$ , glacial  $\text{HC}_2\text{H}_3\text{O}_2$ , or oil of turpentine, but only partly in petroleum benzin. When

one part is shaken with 20 parts of warm water and filtered, the filtrate should give a red colour with a few drops of  $\text{Fe}_2\text{Cl}_6$  reagent 1:1,000; it reduces  $\text{AgNO}_3$  in the cold and Fehling's solution on warming. The aqueous filtrate should give no red colour on addition of a few drops of aniline (wood tar products); another portion is not coloured by  $\text{K}_2\text{CrO}_4$  solution (coal tar products). Agitate 1 c.c. of the oil with 15 c.c. of petroleum benzin, filter, add an equal volume of  $\text{Cu } 2\text{C}_2\text{H}_3\text{O}_2$  solution 1:20, shake and allow to separate. On adding an equal volume of  $\text{Et}_2\text{O}$  to the separated benzin solution no intensely green colour should be given, and not more than a light yellow to brown colour produced (rosin and rosin oil).

*Oleum Cajuputi*.—To be colourless or yellowish instead of "colourless or greenish." Sp.g. 0.912 to 0.925 at  $25^\circ\text{C}$ .;  $\alpha_D$  not exceeding  $-4^\circ$  at  $25^\circ\text{C}$ . Should not respond to the volatile oil heavy metals test.

*Oleum Carui*.—To yield not less than 50 per cent. of carvone. Soluble 1:8 in EtOH 80 per cent. For assay take 10 c.c. of the oil in a Hirschsohn flask with a 10 c.c. graduated neck. Add 50 c.c. of  $\text{Na}_2\text{SO}_3$  solution which has been neutralized by means of a little  $\text{HC}_2\text{H}_3\text{O}_2$  containing phenolphthalein. Heat with agitation in the boiling water-bath, neutralizing the mixture from time to time by adding  $\text{HC}_2\text{H}_3\text{O}_2$ . When no colour appears on adding a few drops more of phenolphthalein and further heating for 15 minutes, the unabsorbed oil is run up into the graduated neck by the addition of more  $\text{Na}_2\text{SO}_3$  solution, and its volume read off. The difference between this and 10 c.c.=carvone.

*Oleum Caryophylli*.—Eugenol, 82 per cent. Sp.g. from 1.038 to 1.060 at  $25^\circ\text{C}$ . Slightly laevorotatory, not exceeding  $-1^\circ 10'$ . In the assay, 50 c.c. of KOH reagent and 10 c.c. of the oil, after shaking for 5 minutes, are heated on the water-bath to complete the reaction.

*Oleum Chenopodii*.—Sp.g. 0.955 to 0.980 at  $25^\circ\text{C}$ .;  $\alpha_D$   $-4^\circ$  to  $-10^\circ$ ; soluble 1:8 in EtOH 70 per cent.

*Oleum Cinnamoni*.—Distilled from young twigs of *Cinnamomum cassia* rectified by steam distillation. (This is the "Cassia oil" of British pharmacy.—Ed. Y.B.) Sp.g. from 1.045 to 1.063;  $\alpha_D$  from  $+1^\circ$  to  $-1^\circ$  at  $25^\circ\text{C}$ . Should not respond to the volatile oil test for heavy metals. Shake 2 c.c. of the oil with 5 to 10 c.c. of petroleum benzin and separate the latter. The solution should be colourless and should not

give a green colour on shaking with an equal volume of 1 : 1,000 Cu  $2\text{C}_2\text{H}_3\text{O}_2$  solution (rosin). Assay for cinnamic aldehyde as for carvone in caraway oil. Should contain 80 per cent. of aldehyde.

*Oleum Coriandri*.—Sp.g. 0.863 to 0.875 at 25°C. ;  $\alpha_D$  from +8° to +13° at 25°C. Soluble 1 : 3 in EtOH 70 per cent.

*Oleum Cubebæ*.—Optical rotation from -20° to -40° at 25°C.

*Oleum Eucalypti*.—To contain not less than 70 per cent. of eucalyptol. Soluble 1 : 4 of EtOH 70 per cent. *Assay for eucalyptol*: Introduce 10 c.c. of the oil into a Hirschsohn flask, add enough of an aqueous solution of resorcinol (1 in 2) to fill the flask about four-fifths full. Shake the mixture thoroughly for 5 minutes and then bring the residual oil into the neck of the flask by the further addition of the same strength resorcinol solution. When the resorcinol solution has become clear (usually after standing for several hours) ascertain the volume of the residual oil. The difference in volume between this residuc and the volume of the original oil multiplied by 10, is equivalent to the percentage, by volume, of the eucalyptol present. When oils are rich in eucalyptol, dilute the 10 c.c. taken for the assay with an equal volume of oil of turpentine, before applying the test, to avoid crystallization in the resorcinol solution.

*Oleum Foeniculi*.— $\alpha_D$  from +12° to +24° at 25°C. Soluble in 8 volumes of EtOH 80 per cent. and 1 volume of EtOH 90 per cent. Congealing point not below 3°C. Proceed as directed under oleum anisi, cooling the oil to 0°C.

*Oleum Hedeomæ*.— $\alpha_D$  from +17° to +28° at 25°C. The EtOH solution to show not more than a slightly acid reaction with litmus.

*Oleum Juniperi*.—Distilled from the ripe fruit. Sp.g. from 0.854 to 0.879 at 25°C. The  $\alpha_D$  varies from 0° to -15° at 25°C. Soluble in 1 : 4 of EtOH 94 per cent. with not more than a slight cloudiness.

*Oleum Lavandulæ*.—Sp.g. from 0.875 to 0.888 at 25°C. The  $\alpha_D$  varies from -1° to -10° at 25°C. Shake 20 c.c. of the oil with 40 c.c. of 5 per cent. EtOH, and when the mixture has cleared withdraw 30 c.c. of the EtOH solution. Neutralize this with N/2 KOH, using phenolphthalein indicator, then add 5 c.c. of N/2 KOH, and heat the mixture on a water-bath under a reflux condenser during 1 hour. Not less than 4.7

c.c. of N/2 HCl is required for neutralization after saponification.

*Oleum Limonis.*—The  $n_{D20} = 1.4744$  to  $1.4755$ ;  $\alpha_{D25}$  from  $+57^{\circ}$  to  $+64^{\circ}$  at  $25^{\circ}\text{C}$ . The first 10 per cent. of the oil, obtained by distillation, as described under *oleum aurantii corticis*, is not more than  $5^{\circ}$  less than that of the original oil. The  $n_D$  of this first 10 per cent. of distillate is not less than  $0.0020$  nor more than  $0.0027$  lower than that of the original oil at  $20^{\circ}\text{C}$ . *Assay for citral*: Introduce about 15 c.c. into a tared 300 c.c. flask, and note the exact weight; add 10 c.c. of a solution of phenylhydrazine (not darker in colour than pale yellow) in alcohol (1 in 10), and allow it to stand for half an hour at room temperature. Then add a few drops of methyl-orange solution and neutralize the liquid exactly with N/2 HCl. If difficulty is experienced in detecting the end point of the reaction, carry the titration until the solution is distinctly acid, transfer it to a separatory funnel and draw off the alcoholic portion. Now wash the oil with distilled water, adding the washings to the EtOH solution, and titrate the latter with N/2 KOH. Carry out a blank test identical with the foregoing except that the oil of lemon is omitted, and note the amount of N/2 HCl consumed. Subtract the number of c.c. of N/2 KOH from the number of c.c. of N/2 HCl used in the original test and this result from the corresponding number of c.c. required in the blank test; each c.c. of this difference corresponds to  $0.076$  Gm. of aldehydes, calculated as citral.

*Oleum Menthae Piperitæ.*—The oil distilled from the flowering herb rectified by steam distillation. Ester content not less than 5 per cent.  $\alpha_D$  from  $-20^{\circ}$  to  $-35^{\circ}$  at  $25^{\circ}\text{C}$ . Solubility in EtOH and reaction to litmus omitted, and "no separation of oil globules" added to solubility in 70 per cent. EtOH. Modified test: Distil about 1 c.c. from 25 c.c. of the oil and pour the distillate on an aqueous solution of  $\text{HgCl}_2$  (1 in 25); a white film does not form at the zone of contact within 1 minute (dimethyl sulphide—found in non-rectified oils). *Assay for esters and total menthol*: Ten c.c. of the original oil is taken for the menthol assay instead of the washed residual oil from the menthyl acetate assay; otherwise the assay remains unchanged.

*Oleum Menthae Viridis.*—Yielding not less than 40 per cent., by volume, of carvone;  $\alpha_D$  from  $-35^{\circ}$  to  $-50^{\circ}$  at  $25^{\circ}\text{C}$ . *Assay for carvone*: As directed for carvone under *oleum carui*.

*Oleum Myristicæ.*—Sp.g. from  $0.859$  to  $0.924$  at  $25^{\circ}\text{C}$ .;  $\alpha_D$

from  $+14^{\circ}$  to  $+30^{\circ}$  at  $25^{\circ}\text{C}$ . Evaporate 3 Gm. of the oil on a water-bath; not more than 0.06 Gm. of residue should remain.

*Oleum Picis Liquidæ*.—Sp.g. from 1.012 to 1.065 at  $25^{\circ}\text{C}$ . Soluble in EtOH, the solution showing an acid reaction with litmus.

*Oleum Pimentæ*.—Sp.g. 1.018 to 1.048 at  $25^{\circ}\text{C}$ .;  $\alpha_D$   $0^{\circ}$  to  $-4^{\circ}$  at  $25^{\circ}\text{C}$ .; soluble 1 : 1 in EtOH 90 per cent. To be assayed for eugenol as described under oleum caryophylli.

*Oleum Pini Pumilionis*.—Distilled from leaves of *Pinus montana*. Sp.g. 0.853 to 0.869 at  $25^{\circ}\text{C}$ . No fraction distils below  $170^{\circ}\text{C}$ .

*Oleum Rosmarini*.—Solubility in EtOH 90 per cent. omitted. Assay as directed under oleum menthæ piperitæ, the oil of rosemary factors of the U.S.P. 1910 remaining unchanged.

*Oleum Santali*.— $\alpha_D$  from  $-15^{\circ}$  to  $-20^{\circ}$  at  $25^{\circ}\text{C}$ . Solubility in EtOH omitted. Assay for santalol: As directed for menthol under oleum menthæ piperitæ in the U.S.P. 1910, using the factors given under sandal oil, but changing 11.026 to 10.926.

*Oleum Sassafras*.—Sp.g. 1.065 to 1.077 at  $25^{\circ}\text{C}$ .;  $\alpha_D$  from  $+3^{\circ}$  to  $+4^{\circ}$ ; soluble 1 : 2 in EtOH 90 per cent. to a neutral solution.

*Oleum Sinapis Volatile*.—To contain not less than 92 per cent. of allyl isocyanide,  $\text{CSNC}_3\text{H}_3$ . Synthetic, or distilled from *Brassica nigra* seeds, previously freed from fatty oil by maceration and subsequent distillation. Optically inactive. Distils completely between  $148^{\circ}$  and  $154^{\circ}\text{C}$ ., the first and last fractions having nearly the same sp.g. as the original oil (EtOH,  $\text{CHCl}_3$ , petroleum and fats). Should give no blue colour with 1 drop of  $\text{Fe}_2\text{Cl}_6$  solution when tested in solution in EtOH (phenols). Assay for allyl isocyanide: Process of U.S.P. 1910 unchanged except that boiling is performed under a reflux condenser.

*Oleum Terebinthinæ Rectificatum*.—To be dried by shaking with  $\text{CaCl}_2$  and filtering. Sp.g. from 0.856 to 0.865 at  $25^{\circ}\text{C}$ . Residue on evaporation not to exceed 0.010 Gm. from 10 c.c.

*Oleum Thymi*.—Sp.g. from 0.894 to 0.929 at  $25^{\circ}\text{C}$ .;  $\alpha_D$  omitted. Soluble 1 : 2 of EtOH 80 per cent. Assay for phenols to be performed in a Hirschsohn flask instead of a burette.

#### CHEMICALS AND GALENICALS.

*Acidum Citricum*.—M.p. omitted. Added tests: An aqueous solution of citric acid (1 : 10), which has been nearly neutralized with AmOH, remains clear on the addition of  $\text{CaSO}_4$  reagent



(oxalic acid). Heat about 5 Gm. of powdered citric acid for 15 minutes on a water-bath with 5 c.c. of  $\text{H}_2\text{SO}_4$  in a porcelain dish, which has been previously rinsed with  $\text{H}_2\text{SO}_4$ , keeping the mixture protected from dust. No darker colour than yellow develops (tartaric acid). Dissolve 10 Gm. of citric acid in 20 c.c. of distilled water, add 2 c.c. of  $\text{H}_2\text{SO}_3$  and boil the mixture until the odour of  $\text{SO}_2$  is barely perceptible. Cool the solution, mix it with 1 c.c. of a solution of  $\text{NaCN}$  in distilled water (1 in 10) and follow this immediately with stronger  $\text{AmOH}$  until the solution possesses a slight odour of  $\text{AmOH}$ . When cold, transfer the solution to a glass-stoppered cylinder of practically colourless glass, graduated at 50 c.c., dilute with sufficient distilled water to measure 50 c.c. and add 3 drops of a solution of  $\text{Na}_2\text{S}$  in distilled water (1 in 10). After mixing the solution well, the colour produced, if any, when viewed downward against a white surface, is not greater than the colour of a solution prepared as follows: To prepare the solution for the blank test, dissolve 3 Gm. of  $\text{AmCl}$  (conforming to the tests for purity described in the Appendix) in 20 c.c. of distilled water, add 4 c.c. of a solution containing 0.080 Gm. of  $\text{Pb}_2\text{NO}_3$  in 1,000 c.c. of distilled water, and then 1 c.c. of diluted  $\text{HCl}$ . Treat this solution with  $\text{H}_2\text{SO}_3$ ,  $\text{NaCN}$  and stronger  $\text{AmOH}$ , then dilute and mix with  $\text{Na}_2\text{S}$  as described above. Before adding the sodium sulphide solution, the liquid must possess a distinct odour of ammonia. The two cylinders used must be matched and must be of practically colourless glass and have the same internal diameter (lead).

*Acidum Hydriodicum Dilutum.*—Rubric changed from “not less than 10 per cent.” to “not less than 9.5 per cent. nor more than 10.5 per cent.” Added test: Mix 0.5 c.c. of diluted hydriodic acid with 10 c.c. of distilled water, add 8 c.c. of  $\text{AgNO}_3$  and 6 c.c. of ammonium carbonate solutions, digest the mixture for 10 minutes on a bath of boiling water, cool, and filter. The filtrate, upon supersaturating with  $\text{HNO}_3$ , should not become more than slightly opalescent (chloride). Residue of 8.3 per cent. on evaporation changed to 3 per cent. on evaporation and ignition at low red heat.

*Acidum Hydrobromicum Dilutum.*—Rubric changed from “not less than 10 per cent.” to “not less than 9.5 per cent. nor more than 10.5 per cent.” Residue on evaporation changed from “no appreciable residue from 10 c.c.” to “not more than 0.0025 Gm. from 25 c.c.” *Assay:* Weigh

accurately about 20 c.c. of diluted hydrobromic acid, dilute it with 30 c.c. of distilled water, and titrate with N/KOH, using methyl-orange indicator.

*Acidum Tartaricum* is directed to be tested for lead by a test similar to that given for citric acid.

*Aether*.—Ether for anæsthetic purposes to be packed in small containers, and not to be used as an anæsthetic after these have been opened for 24 hours. Sp.g. from 0.713 to 0.716 at 25°C. B.p. about 35°C. The moist residue left on spontaneous evaporation of 25 c.c. of ether from a shallow dish is odourless, and neither reddens nor bleaches blue litmus paper, and dried at 100°C. does not exceed 0.001 Gm. On shaking 10 c.c. of ether occasionally for 1 hour with 1 c.c. of KOH reagent in a glass-stoppered tube protected from light no colour should be developed in either liquid (aldehyde). Added test: Shake 10 c.c. of ether for 1 hour with 1 c.c. of freshly made KI and  $\text{CaI}_2$  solution 1:10, in a stoppered cylinder, previously rinsed with the ether; no colour should be developed in either liquid if protected from light (peroxides). The test for undue amount of water or of EtOH to be omitted.

*Aqua Hamamelidis*.—Process to be omitted. Neutral or only faintly acid. Sp.g. 0.979 to 0.982 at 25°C. Free from mucilaginous or fungus growths or an acetous odour. Gives no reaction with  $\text{H}_2\text{S}$  or with AmHS solutions. Not more than 0.025 Gm. of residue from evaporation of 100 c.c. Add 8 drops of an aqueous solution of resorcinol (1 in 200) to 5 c.c. of hamamelis water, and then carefully pour this upon 5 c.c. of  $\text{H}_2\text{SO}_4$  in such a manner that the two liquids do not mix. After standing 3 minutes a rose-red ring should not appear at the line of contact of the liquids nor should a distinct, white layer appear above this zone (formaldehyde). Ten c.c. of hamamelis water should give no reaction for MeOH when treated according to the test given under alcohol for the detection of MeOH.

*Aspidosperma*.—The dried bark of *Aspidosperma quebracho-blanco* without admixture of more than 2 per cent. of wood or other foreign matter. A full description of macro- and microscopic characters is given.

*Calcii Glycerophosphas*.—To contain not less than 90 per cent. of anhydrous normal calcium glycerophosphate. A fine white, odourless and almost tasteless, somewhat hygroscopic powder. One Gm. dissolves in about 50 c.c. of water at 25°C.; soluble in less water at a lower temperature; the presence of citric acid in-

creases its solubility ; insoluble in alcohol. An aqueous solution is alkaline to litmus and to phenolphthalein. A cold, saturated, aqueous solution yields white, iridescent scales of anhydrous calcium glycerophosphate when heated to boiling. When heated above  $170^{\circ}\text{C}$ . the salt is decomposed, evolving inflammable vapours and at a red heat is converted into calcium pyrophosphate. A saturated aqueous solution of the salt yields with  $\text{Am}_2\text{C}_2\text{O}_4$  reagent a white precipitate, insoluble in  $\text{HC}_2\text{H}_3\text{O}_2$  but soluble in  $\text{HCl}$ . With  $\text{Pb}_22\text{C}_2\text{H}_3\text{O}_2$  reagent the saturated solution yields a white precipitate which is soluble in  $\text{HNO}_3$ . Dissolve 1 Gm. of calcium glycerophosphate in 10 c.c. of diluted  $\text{HNO}_3$  and add an equal volume of cold ammonium molybdate reagent ; no precipitate should be formed within one hour (phosphates). On heating the mixture, however, a yellow precipitate will be formed. Ten c.c. of an aqueous solution of the salt (1 in 100) in water, acidified with a few drops of  $\text{HCl}$ , does not respond to the test for heavy metals. Dissolve 0.1 Gm. of the salt in 10 c.c. of diluted  $\text{HNO}_3$  and add 1 c.c. of  $\text{AgNO}_3$  reagent ; a distinct opalescence may appear but no precipitate within one minute (chloride). Dissolve 0.1 Gm. of the salt in 10 c.c. of diluted  $\text{HCl}$  and add 1 c.c. of  $\text{BaCl}_2$  reagent ; no distinct turbidity appears within one minute (sulphate). Shake 1 Gm. of finely powdered calcium glycerophosphate with 25 c.c. of absolute  $\text{EtOH}$ , filter the mixture, evaporate the filtrate on a water-bath and dry the residue for an hour at a temperature not exceeding  $70^{\circ}\text{C}$ . This residue does not weigh more than 0.01 Gm. (limit of alcohol-soluble impurities). Weigh accurately from 0.5 to 1 Gm. of the finely powdered salt and dry it to constant weight at  $130^{\circ}\text{C}$ . ; the loss does not exceed 10 per cent. (limit of water). Weigh accurately about 0.4 Gm. of the salt, previously dried to constant weight at  $130^{\circ}\text{C}$ ., dissolve it in 20 c.c. of a 5 per cent. solution of  $\text{HC}_2\text{H}_3\text{O}_2$  and add 30 c.c. of distilled water. Heat the mixture to boiling and add an excess of  $\text{Am}_2\text{C}_2\text{O}_4$  reagent. Collect the resulting precipitate, wash, dry, and then ignite it until of constant weight. This residue of  $\text{CaO}$  weighs not less than 23.47 per cent. of the weight of calcium glycerophosphate taken. Weigh accurately about 1 Gm. of calcium glycerophosphate and ignite it to constant weight ; the resulting residue of calcium pyrophosphate weighs not less than 54.4 per cent. of the amount taken.

*Ceratum*.—White petrolatum omitted ; new formula : white wax, 300 Gm. ; benzoated lard, 700 Gm.

*Ceratum Cantharidis*.—Cantharides, in No. 60 powder, 350 Gm. ; glacial acetic acid, 25 c.c. ; oil of turpentine, 150 c.c. ; yellow wax, 175 Gm. ; rosin, 175 Gm. ; benzoated lard, 200 Gm. To make 1,000 Gm. Macerate the cantharides for 48 hours in a warm place, in a covered container, with the mixed oil of turpentine and glacial acetic acid. Melt together the rosin, yellow wax and lard, add the macerated cantharides and heat the mixture on a water-bath, with occasional stirring, until it weighs 1,000 Gm. Finally stir until firm. Formerly 320 Gm. of cantharides were macerated with 150 Gm. of liquid petrolatum for 48 hours under the same conditions, the mixture was then added to the melted rosin, wax and lard and heated for one hour on a water-bath before cooling.

*Collodium Flexile*.—New formula : Collodion, 950 Gm. ; camphor, 20 Gm. ; castor oil, 30 Gm. To make 1,000 Gm., weigh the ingredients, successively, into a tared bottle and shake the mixture until the camphor is dissolved.

*Cresol*.—Modified definition : A mixture of the isomeric cresols ( $C_6H_4.CH_3OH$ ) obtained from coal tar. Sp.g. from 1.030 to 1.038 at 25°C. One c.c. of cresol dissolves in about 50 c.c. of water, usually forming a cloudy solution ; it is miscible with EtOH, Et<sub>2</sub>O, C<sub>6</sub>H<sub>6</sub>, petroleum benzin, and glycerin ; it is soluble in solutions KOH or NaOH. A saturated aqueous solution of cresol gives a blue-violet reaction with Fe<sub>2</sub>Cl<sub>6</sub> reagent and is neutral or shows a slight acid reaction to litmus. A solution of 1 c.c. in water 60 c.c. shows not more than a slight turbidity.

*Elixir Adjvans*.—Title changed to *Elixir Glycyrrhizæ*.

*Emplastrum Belladonnæ*.—Process omitted. A rosin plaster or rubber adhesive plaster base permitted. To contain 30 per cent. of extract of belladonna leaves, and to yield not less than 0.35 nor more than 0.40 per cent. of mydriatic alkaloids. Method of assay : Remove the cloth from the face of the plaster and introduce 10 Gm. of the spread material cut into strips into a flask, add 50 c.c. of CHCl<sub>3</sub> and shake until the plaster is dissolved. Pour off the CHCl<sub>3</sub> solution and wash the cloth with 25 and 25 c.c. of CHCl<sub>3</sub>, adding these washings to the first solution. Then wash the cloth with another 80 c.c. of CHCl<sub>3</sub> containing 1 c.c. of AmOH solution. Add to the previously bulked CHCl<sub>3</sub> solutions. Stir gently and allow to stand until the rubber has settled into a compact mass. Dry the washed cloth and weigh it ; subtracting its weight from the original weight taken.

Transfer the  $\text{CHCl}_3$  solution to a separator, rinsing the container with 10 c.c. of  $\text{EtOH}$  and add this to the  $\text{CHCl}_3$ . Then add 100 c.c. of water, rotate until mixed and allow to separate. Draw off the  $\text{CHCl}_3$  into a second separator containing 50 c.c. of water, shake out, separate the  $\text{CHCl}_3$ , and put back the aqueous portion into the first separator. Again put back the  $\text{CHCl}_3$  into the second separator. Shake out the aqueous liquid in the first separator with 10 and 5 c.c. of  $\text{CHCl}_3$ , adding them to the  $\text{CHCl}_3$  in the second. Completely extract the alkaloids in this by shaking out with successive quantities of water containing dilute  $\text{H}_2\text{SO}_4$ . Collect the acid washings in a separator, make alkaline with  $\text{AmOH}$  and shake out with  $\text{CHCl}_3$ . Filter the  $\text{CHCl}_3$  through cotton, evaporate the solvent, dissolve the residue in 5 c.c. of  $\text{N}/10 \text{ H}_2\text{SO}_4$  and titrate back with  $\text{N}/50 \text{ KOH}$  solution with cochineal indicator. Each c.c. of  $\text{N}/10 \text{ H}_2\text{SO}_4$  used up = 0.02892 Gm. of mydriatic alkaloids.

*Emplastrum Cantharidis*.—Cantharides cerate, rosin plaster, of each, q.s. Prepare the plaster by spreading the cerate on rosin plaster, leaving a margin round the edges. Each square cm. so spread is to contain 0.1 Gm. of the cerate. It may also be spread on other suitable material. It should be made extemporaneously.

*Emplastrum Plumbi*.—Lead oxide, 1,000 Gm.; olive oil, 1,000 Gm.; lard oil, 1,000 Gm.; boiling water, q.s. Heat the oils in a Cu vessel of the capacity at least 4 times that of the bulk of ingredients. Sift the  $\text{PbO}$  on the surface of the hot oil and mix thoroughly. Then add the water gradually and continue boiling, stirring constantly with a wooden spatula, keeping up the volume of water evaporated; boil until a homogeneous mass results. Then remove from the fire and wash the mass several times with warm water to remove glycerin. Finally knead free from water, roll into suitable sizes and wrap in paraffin paper.

*Emplastrum Resinæ*.—Rosin in fine powder, 140 Gm.; lead plaster, 800 Gm.; yellow wax, 60 Gm. Melt the lead plaster and wax; add the rosin; when melted, strain; cool and stir until it stiffens.

*Extractum Physostigmatis Pulveratum*.—Yields not less than 1.7 per cent. nor more than 2.3 per cent. of alkaloids. One Gm. of powdered extract = 13 Gm. of average physostigma. Exhaust 1,000 Gm. of the drug in No. 60 powder, first using 1,000 c.c. of a mixture of alcohol 94 per cent. 3 vols. and water 1 vol.

in which tartaric acid 5 Gm. is dissolved. Continue the percolation with the same menstruum without acid, until exhausted. Distil off the alcohol at as low a temperature as possible, and evaporate at not above 80°C to 200 c.c. Shake out this residue with 250 and 200 c.c. of petroleum benzin, reject these washings, and evaporate the aqueous portion at 80°C. to pilular consistence. Incorporate 20 Gm. of dry starch and dry in warm air. Powder, assay, and dilute with required quantity of starch.

*Extractum Viburni Prunifolii Pulveratum.*—One Gm. of powdered extract = 5 Gm. of drug. Exhaust the drug in No. 30 powder with alcohol 48·6 per cent. Distil off the EtOH, evaporate at 80°C. Incorporate 5 Gm. of MgO and dry on glass plates in warm air. Powder and add sufficient starch to make 200 Gm.

*Fluid Extractum Cascaræ Sagradæ Aromaticum.*—Cascara powder, 1,000 Gm. ; MgO, 125 Gm. ; extract of licorice, 40 Gm. ; glycerin, 200 c.c. ; alcohol 94 per cent., 250 c.c. ; benzosulphinide (gluside), 1 Gm. ; oil of anise, 2·5 c.c. ; oil of cassia, 0·2 c.c. ; oil of coriander, 0·1 c.c. ; oil of betula, 0·2 c.c. ; boiling water, a sufficient quantity, to make 1,000 c.c. Mix the cascara sagrada with the MgO, moisten with 2,000 c.c. of boiling water, stirring occasionally during 2 hours, and percolate with boiling water until exhausted. Evaporate percolate to 500 c.c., and while warm, dissolve in it the extract of licorice. When cold, add the glycerin, then the alcohol containing the benzosulphinide and the oils, and then add sufficient water to make 1,000 c.c.

*Fluid Extractum Scillæ.*—Macerate 1,000 Gm. of squill, in No. 20 powder, for 2 hours, with sufficient of a mixture of 2,000 c.c. alcohol 94 per cent., 1,000 c.c. water in a tightly-covered vessel. Then shake down evenly in a percolator, add more of the same menstruum and, when saturated, macerate 48 hours. Now percolate slowly, using same menstruum, to obtain 1,000 c.c. of percolate. Again macerate drug in percolator for 12 hours, afterwards collecting a second 1,000 c.c. of percolate. Again macerate for 12 hours and collect a third percolate of 3,000 c.c. Distil the EtOH from the mixed percolates at as low a temperature as possible, and evaporate the liquid to 800 c.c. Slowly add to this residue when cold, with continuous agitation, 2,000 c.c. of alcohol 94 per cent. and set aside, tightly closed for 12 hours. Decant supernatant liquid from syrupy layer, filter decanted liquid and wash syrupy residue with two portions, 300 c.c. each, of a mixture of alcohol 94 per cent. 4 vols., water 1 vol., passing the washings through the filter into the previously col-

lected alcoholic liquid. Reduce the combined alcoholic liquids, by distillation, to 800 c.c. and add alcohol 48.6 per cent. to make 1,000 c.c.

*Gelatinum.*—Modified description: An amorphous solid, in sheets, flakes, ground, powdered or shredded form, colourless or slightly yellowish, and having a slight characteristic odour and taste; unalterable in the air when dry, but decomposing when moist or in solution. Modified test: A hot solution of gelatin in distilled water (1 in 40) should be free from putrid odour, and is not more than slightly acid to litmus; it appears not more than slightly opalescent in a stratum of 2 cm. and on cooling to 6°C. and standing for several hours it forms a firm, transparent or translucent jelly. Ash changed from 2 per cent. to "not more than 3 per cent." Added tests: A solution of the ash in 25 c.c. of distilled water, made with the aid of heat and a few drops of HCl, does not respond to the test for heavy metals. Heat 1.5 Gm. of gelatin with 30 c.c. of HCl (1 in 4) in a flask on a water-bath, and when dissolved, add 3 c.c. of saturated Br water and heat it on a water-bath for 15 minutes, shaking the flask occasionally. Then add 0.5 Gm. of KI and follow it immediately with 0.5 c.c. of a 25 per cent. solution of  $\text{SnCl}_2$ . Heat the solution for 5 minutes on a water-bath, cool and subject it to the test for As. The stain produced, if any, is not greater than that produced in a test made with the same quantities of the reagents to which 2 c.c. of the standard  $\text{As}_2\text{O}_3$  solution has been added.

*Glucosum.*—The product obtained by the hydrolysis of starch, consisting chiefly of dextrose and dextrins. A colourless or slightly coloured, thick, syrupy liquid. Very soluble in water; sparingly soluble in alcohol 94 per cent. The aqueous solution is neutral or slightly acid to litmus. Gives a red precipitate with Fehling's reagent, hot. Weigh accurately about 0.5 Gm. of the sample in a tared wide glass-stoppered weighing bottle, add 2 c.c. of water, evaporate at 70°C., then dry to constant weight at 90°C. The loss of weight should not exceed 21 per cent. Ash not above 1 per cent. A solution of 5 Gm. in 15 c.c. of distilled water should not require more than 0.6 c.c. of N/10 KOH to give a pink colour with phenolphthalein indicator (limit of free acid). Two Gm. of glucose in 50 c.c. of water boiled for one minute should give no blue colour with one drop of I reagent (starch). On now adding a few drops of starch reagent, a blue colour should be produced ( $\text{SO}_2$ ). Ten c.c. of a

5 per cent. solution of glucose should give no reaction for heavy metals. If this solution is not colourless comparison should be made with 10 c.c. of the same solution, to which a volume of water, equal to the  $\text{H}_2\text{S}$  solution used in the test, has been added. Dissolve 1.5 Gm. of glucose in 5 c.c. of water, add 5 c.c. of dilute  $\text{H}_2\text{SO}_4$  and 1 c.c. of Br water. Heat for 5 minutes on the water-bath. Then add 0.5 Gm. of KI, followed by 5 drops of  $\text{SnCl}_2$  reagent, cool and test for As. The stain produced should not exceed that given with a blank plus 2 c.c. of a standard As solution.

*Glyceritum Hydrastis*.—New assay: 100 c.c. yields not less than 1.12 Gm. nor more than 1.39 Gm. of  $\text{Et}_2\text{O}$ -soluble alkaloids. Proceed as directed in assay of fluid extract of belladonna, using 5 c.c. of the glycerite and only  $\text{Et}_2\text{O}$  as the immiscible solvent. Wash the final  $\text{Et}_2\text{O}$  extract with 10 c.c. of water, and reject this aqueous washing. Filter the  $\text{Et}_2\text{O}$  solution through cotton, wash the cotton with  $\text{Et}_2\text{O}$ , evaporate the filtrate and washings, dry the residue at  $100^\circ\text{C}$ . and weigh.

*Magma Bismuthi*.—Each 100 c.c. should contain an amount of bismuth hydroxide equivalent to not less than 5.5 Gm. nor more than 6 Gm. of  $\text{Bi}_2\text{O}_3$ .  $\text{BiON}(\text{O})_2\text{H}_2\text{O}$ , 80 Gm.;  $\text{HNO}_3$ , 120 c.c.;  $\text{Am}_2\text{CO}_3$ , 10 Gm.;  $\text{AmOH}$ , 10 per cent.; water and distilled water, of each, q.s. to make 1,000 c.c. Dissolve the bismuth subnitrate in 60 c.c. of distilled water and 60 c.c. of nitric acid by warming gently. Pour this solution, with constant stirring, into 5,000 c.c. of distilled water to which 60 c.c. of  $\text{HNO}_3$  has been added. Dilute 480 c.c. of  $\text{AmOH}$  with 4,000 c.c. of distilled water in a capacious vessel. Dissolve the  $\text{Am}_2\text{CO}_3$  in this solution and then quickly pour the bismuth solution into it with constant stirring. If the mixture is not distinctly alkaline, add a sufficient quantity of  $\text{AmOH}$  to make it so, allow the precipitate to subside, decant the supernatant liquid and wash the precipitate twice with distilled water, by decantation. Transfer the magma to a strainer of close texture, arranged in a percolator so as to provide continuous washing with distilled water, the outlet tube being elevated to prevent the surface of the magma from becoming dry; allow the operation to proceed until the washings cease to react with phenolphthalein. Transfer the moist magma to a graduated vessel and add a sufficient quantity of distilled water to make the product measure 1,000 c.c. and mix thoroughly. The product should be neutral to litmus and phenolphthalein. One c.c. of  $\text{HCl}$  added to 1 c.c. of bismuth magma produces a



clear solution. Pour the clear solution into 10 volumes of distilled water; a white precipitate is produced. Evaporate 10 c.c. of bismuth magma to dryness and ignite the residue to constant weight; not less than 0.550 Gm. nor more than 0.60 Gm. of  $\text{Bi}_2\text{O}_3$  results.

*Massa Ferri Carbonatis*.—*Added assay*: Weigh accurately about 1 Gm. of mass of ferrous carbonate; dissolve it in 15 c.c. of diluted  $\text{H}_2\text{SO}_4$  and dilute the solution with distilled water to about 100 c.c. The immediate titration  $\text{N}/10 \text{ K}_2\text{Cr}_2\text{O}_7$  with  $\text{K}_6\text{Fe}_2\text{Cy}_{12}$  indicator, shows not less than 41.5 per cent. of  $\text{Fe}_2\text{CO}_3$ .

*Massa Hydrargyri*.—*Added assay*: Weigh accurately about 1 Gm. of mass of mercury, dissolve it in a mixture of 10 c.c. of distilled water and 5 c.c. of  $\text{HNO}_3$ ; heat on a water-bath until red fumes cease to be evolved, and the liquid becomes pale yellow. Add 150 c.c. of distilled water and 2 c.c. of ferric ammonium sulphate reagent and titrate the solution with  $\text{N}/10 \text{ KCNS}$ . It shows not less than 32 per cent. nor more than 34 per cent. of Hg.

*Nitrogenii Monoxidum*.—Quite soluble in water at low temperatures; at  $25^\circ\text{C}$ . 1 volume of water dissolves about 1.3 volumes of  $\text{N}_2\text{O}$ . Pass 2,000 c.c. of the gas, measured under normal atmospheric pressure at about  $25^\circ\text{C}$  into 100 c.c. of  $\text{Ba}(\text{OH})_2$  solution—at a rate not exceeding 4,000 c.c. per hour; not more than a slight turbidity is produced ( $\text{CO}_2$ ). No opalescence is produced in a mixture of 100 c.c. of distilled water and 1 c.c. of  $\text{AgNO}_3$  reagent by 2,000 c.c. of the gas under the conditions described above (halogens). No change in colour is produced in 100 c.c. of distilled water to which 5 drops of litmus have been added by the passage of 1,000 c.c. of the gas as above (acids or bases). The colour of a solution 0.2 c.c. of  $\text{N}/10 \text{ KMnO}_4$  solution in 100 c.c. is not affected by 1,000 c.c. of the gas treated in the same way.

*Oleoresina Petroselinii*.—The ether extract of parsley fruit, obtained by percolating parsley fruits with ether and distilling off the solvent. After standing for 4 or 5 days, the clear liquid portion is decanted from any solid residue.

*Oleum Sesami*.—Sesame oil is suggested for inclusion.

*Oleum Terebinthinæ*.—The following alterations in characters are suggested. Optical rotation variable; soluble 1 : 5 in alcohol 94 per cent.; residue not more than 0.05 Gm. from 10 Gm. Distil 200 c.c. from a 300 c.c. distilling flask, with side tube 8 cm. above the globe, at rate of 2 drops a second; 90 per cent. of the oil distils between  $154^\circ$  and  $170^\circ\text{C}$ ., the temperature

being read with the column of mercury immersed in the vapour. Five c.c. of oil shaken with an equal volume of HCl does not give a brownish or greenish colour. Introduce 5 c.c. of oil of turpentine drop by drop into a 50 c.c. flask with a long graduated neck and containing 25 c.c. of fuming sulphuric acid. Agitate the mixture cautiously for 5 minutes, keeping the temperature just below 65° by immersion in cold water. Then cool and add  $\text{H}_2\text{SO}_4$  to fill up to the higher graduation. The clear reddish viscous layer which separates after the dark mass has settled for 2 hours should not exceed 1 per cent. A larger volume of colourless oil with a  $\eta_{D20}$  of less than 1,500 indicates the presence of mineral oil.

*Pilule Ferri Carbonatis*.—*Added assay*: Dissolve 3 pills in 15 c.c. of diluted  $\text{H}_2\text{SO}_4$  and dilute with distilled water to about 100 c.c. The immediate titration with N/10  $\text{K}_2\text{Cr}_2\text{O}_7$  with  $\text{K}_6\text{Fe}_2\text{Cy}_{12}$  indicator, shows not less than 0.065 Gm. of  $\text{FeCO}_3$  in each pill.

*Pilule Ferri Iodidi*.—*Added assay*: Dissolve 5 pills in 15 c.c. of diluted  $\text{H}_2\text{SO}_4$  and dilute with distilled water to about 100 c.c. The immediate titration with N/10  $\text{K}_2\text{Cr}_2\text{O}_7$  as above, shows not less than 0.04 Gm. of Fe in each pill.

*Potassii Chloras*.—*Modified test*: Ten c.c. of an aqueous solution of the salt (1 in 20) does not respond to the test for heavy metals. *Modified assay*: Weigh accurately about 0.1 Gm. of  $\text{KClO}_3$ , transfer it to a 250 c.c. flask and dissolve it in 10 c.c. of distilled water. Then add 25 c.c. of acidulated  $\text{FeSO}_4$  reagent, insert a valve stopper (see below) and boil the mixture for 10 minutes. Now cool the mixture, add 10 c.c. of a 10 per cent.  $\text{MnSO}_4$  solution and titrate the excess of  $\text{FeSO}_4$  with N/10  $\text{KMnO}_4$ . At the same time conduct a parallel experiment with another portion of 25 c.c. of acidulated  $\text{FeSO}_4$  to ascertain the total amount of  $\text{FeSO}_4$  in the solution used. *Valve Stoppers*.—Take a piece of rubber tubing of convenient diameter and about 5 cm. in length and, having placed a piece of glass rod in one end and having slipped the other end over a glass tube which passes through a perforated stopper of a size convenient to fit the flask used, cut a longitudinal slit about 15 mm. long in one side of the rubber tube about half way up.

*Resina*.—Sp.g. changed to "from 1.07 to 1.09" at 25°C. Ash statement changed to "ash not exceeding 0.05 per cent."

*Added description*: Its alcoholic solution shows an acid reaction.

*Scopolaminæ Hydrobromidum*.—Hyoscine hydrobromide added as a synonym. *Modified description*: "The hydrobromide

of laevorotatory scopolamine, also known as hyoscyne, obtained from various plants of the Solanaceæ. Colourless, transparent, odourless, slightly efflorescent, rhombic crystals, sometimes of large size. Its aqueous solution (1 in 20) is neutral or at most only slightly acid to litmus. When anhydrous it melts between  $190^{\circ}$  and  $192^{\circ}\text{C.}$ ;  $\alpha_D + 22^{\circ}$  to  $+25.75^{\circ}$  when determined in an aqueous solution containing the equivalent of 5 Gm. of anhydrous scopolamine hydrobromide in 100 c.c. of solution. at  $25^{\circ}\text{C.}$ , is from  $22^{\circ}$  to  $25.75^{\circ}$ . *Added tests*: Two c.c. of  $\text{CHCl}_3$  shaken with 1 c.c. of an aqueous solution of the salt (1 in 20) to which a few drops of  $\text{Cl}$  water have been added, will cause the  $\text{CHCl}_3$  to assume a brownish colour. When dried to constant weight at  $100^{\circ}\text{C.}$  the loss does not exceed 13 per cent. It also loses its water of crystallization slowly over  $\text{H}_2\text{SO}_4$ . On incinerating 0.1 Gm. no weighable ash remains. A few drops of  $\text{AmOH}$  solution added to 1 c.c. of an aqueous solution (1 in 20) causes no turbidity; the addition of  $\text{KOH}$  reagent, only a whitish, transient turbidity (foreign alkaloids). Add 0.05 c.c. of  $\text{N}/10 \text{ MKnO}_4$  solution to 15 c.c. of an aqueous solution (1 in 100); the solution is not completely decolorized within 5 minutes (apo-atropine). *Modified test*: The solution of about 0.1 Gm. of the salt in 1 c.c. of  $\text{H}_2\text{SO}_4$  produces not more than faint yellow colour (carbonizable impurities); a drop of  $\text{HNO}_3$  added to this solution will produce an orange colour, due to the liberation of bromine, but no deep-red colour, fading to orange, should be noticeable (morphine). The  $\text{PtCl}_4$  test is omitted.

*Serum Preparatum.*—*Added tests*: One Gm. of prepared suet dissolved in 50 c.c. of hot  $\text{EtOH}$  and a few drops of phenolphthalein solution added, does not require more than 0.6 c.c. of  $\text{N}/10 \text{ KOH}$  to produce a pink colour (limit of free acid). Saponification value: not less than 193 nor more than 200. Iodine value: not less than 33 nor more than 48.

*Sodii Glycerophosphas.*—The sodium salt of glycerophosphoric acid containing not less than 66 per cent. of anhydrous sodium glycerophosphate. It occurs either in the form of white, monoclinic plates or scales, as a white powder, or as a semi-solid mass having a saline taste; odourless. Very soluble in cold and hot water, nearly insoluble in  $\text{EtOH}$ . An aqueous solution (1 in 20) shows an alkaline reaction with litmus and a very slightly alkaline reaction with phenolphthalein. Heated to about  $60^{\circ}\text{C.}$  the salt begins to lose water. When strongly heated it is decomposed, evolving inflammable vapours, and at a red heat is con-

verted into sodium pyrophosphate. An aqueous solution (1 in 50), acidified with HCl, does not respond to the test for heavy metals. Dissolve 1 Gm. of sodium glycerophosphate in 20 c.c. of diluted HNO<sub>3</sub> and add an equal volume of cold ammonium molybdate reagent. No precipitate is formed within one hour (phosphates). On heating the mixture, a yellow precipitate will be formed. Triturate about 1 Gm. of sodium glycerophosphate, accurately weighed, with 25 c.c. of absolute alcohol, filter, evaporate the filtrate on a water-bath and dry the residue for one hour at a temperature not exceeding 70°C. The residue weighs not more than 1 per cent. of the amount of salt taken (limit of alcohol-soluble impurities). Weigh accurately about 2.5 Gm. of the salt, dissolve it in 50 c.c. of distilled water and titrate with N/2 HCl, methyl orange as indicator. It indicates not less than 66 per cent. of anhydrous sodium glycerophosphate.

*Syrupus Acaciæ*.—The syrup is to be heated at boiling for 15 minutes, the volume lost made up with boiling water and the product preserved in sterilized bottles.

*Syrupus Calcii Lactophosph.*—Sugar reduced to 650 Gm. and 50 c.c. of glycerin added.

*Syrupus Hypophosphitum*.—Sugar reduced to 600 Gm. Glycerin and added tincture of lemon omitted. Alternative percolation method omitted.

*Syrupus Picis Liquidæ*.—Preliminary washing of tar omitted. Alternative-percolation method included.

*Syrupus Pruni Virginianæ*.—Sugar increased to 800 Gm. Glycerin reduced to 50 c.c. The drug is moistened with water containing the glycerin, allowed to macerate for 24 hours before percolation and 500 c.c. of percolate collected. Sugar is dissolved in this by agitation and water added to make up to 1,000 c.c.

*Syrupus Scillæ Comp.*—The tartarated antimony is dissolved in 10 c.c. of hot water and added to 750 c.c. of syrup, to which the mixed fluid extracts are gradually added and finally enough syrup to make 1,000 c.c.

*Tinctura Cantharidis*.—The drug is macerated with the alcohol under a tube condenser at 50° to 55°C. for 24 hours, with frequent agitation, then percolated to produce the required volume.

*Terra Silicea Purificata*.—(Purified Kieselguhr). The frustules and fragments of diatoms, purified by boiling with diluted HCl, washing and calcining. It does not contain more than 10 per cent. of hygroscopic moisture. Preserve it in tightly closed containers. Purified siliceous earth is a very bulky and very

fine powder, white or of a pale light grey or pale buff colour, without odour or taste. It readily absorbs moisture and will retain about four times its weight of water without the mixture becoming fluid. It is insoluble in water, acids or dilute alkaline solutions. Boil 10 Gm. with 50 c.c. of distilled water and filter the mixture; the filtrate is colourless and neutral to litmus. When ignited it does not darken nor lose more than 10 per cent. of its weight. Add 1 Gm. of purified siliceous earth to 25 c.c. of HCl; no effervescence should occur, and after boiling and filtering, the filtrate is colourless, and separate portions, when tested, yield no precipitate with  $\text{BaCl}_2$  and no blue colour with  $\text{K}_4\text{FeCy}_6$ . Treat 1 Gm. of purified siliceous earth with 20 c.c. of diluted HCl and filter. Ten c.c. of the filtrate, when evaporated to dryness and the residue ignited, should not leave a residue weighing more than 0.005 Gm.

*Tinctura Iodi*.—The 50 Gm. of KI is dissolved in 50 c.c. of distilled water in a bottle, 70 Gm. of I is then dissolved in this solution by agitation and enough EtOH added to make 1,000 c.c. No water was used in the former process.

*Tinctura Sanguinarie*.—Ten c.c. of HCl replaces the 20 c.c. of  $\text{HC}_2\text{H}_3\text{O}_2$ , otherwise the process remains the same.

*Trioxymethylene*.—It contains not less than 96 per cent. of trioxymethylene or paraformaldehyde  $(\text{HCOH})_3$ , a polymeric form of formaldehyde. It occurs in white, friable masses, or as a powder, having a slight odour of formaldehyde. On heating it is partly converted into formaldehyde and partly sublimed unchanged. Slowly soluble in cold water, more readily soluble in hot water with the formation of formaldehyde, insoluble in alcohol or ether; soluble in fixed alkali solutions. A mixture of about 0.01 Gm. each of trioxymethylene and morphine sulphate and 10 drops of  $\text{H}_2\text{SO}_4$  assumes a violet-red colour, changing to blue. Ash not to exceed 0.1 per cent. When 0.5 Gm. is shaken with 10 c.c. of distilled water the latter should be neutral to litmus. *Assay*: Weigh 1 Gm. of trioxymethylene, finely powdered, mix in a flask with 50 c.c. of N/KOH and add through a small funnel 50 c.c. of  $\text{H}_2\text{O}_2$  solution previously neutralized with NaOH. When reaction ceases, wash down the funnel and the sides of the flask with distilled water. Allow to stand for 30 minutes, then titrate the excess of alkali with N/ $\text{H}_2\text{SO}_4$  with litmus indicator.

*Unguentum Acidi Borici*.—The hard paraffin reduced from 100 to 50 Gm. and the white petrolatum correspondingly increased.

*Unguentum Diachylon.*—White petrolatum to replace olive oil.

*Unguentum Hydrarg. Dilut.*—Mercury reduced from 33·5 to 30 per cent.

*Unguentum Hydrarg. Nit.*—Mercury, 7 Gm. ; nitric acid, 17·5 Gm. ; lard, free from water, 76 Gm. Warm the lard in a capacious dish until just melted (about 45°C.), add 7 Gm. of  $\text{HNO}_3$  all at once and continue heating until reaction is complete. Withdraw the heat immediately after the rapid rise of froth and cool, stirring until it assumes a bright yellow colour. Dissolve the Hg in the rest of the  $\text{HNO}_3$ , warming if necessary, and mix the solution with the base.

*Unguentum Phenolis.*—2·25 Gm. of liquefied phenol replaces 3 Gm. of phenol ; and simple ointment is the basis instead of white petrolatum.

**ARSENIC TEST.**—*Standard Arsenic Solution.*—Dissolve 0·1 Gm. of pure  $\text{As}_2\text{O}_3$ , previously dried in a desiccator and accurately weighed, in about 5 c.c. of NaOH solution 1 : 5. Neutralize with dilute  $\text{H}_2\text{SO}_4$ , then dilute to exactly 1,000 c.c., using recently boiled distilled water, at 25°C., to which add 10 c.c. of dilute  $\text{H}_2\text{SO}_4$ . [Obviously all the chemicals used must be As-free.] Take exactly 10 c.c. of this and again dilute to exactly 1,000 c.c. with acidified water at 25°C. as before. One c.c. of this second dilution = 0·001 Mgm. of  $\text{As}_2\text{O}_3$ . A fresh solution should be made when new standard stains are required.

*Preparation of Substance to be Tested.*—Add 1 c.c. of a mixture of equal volumes of strong  $\text{H}_2\text{SO}_4$  and distilled water to 5 c.c. of an aqueous solution 1 : 25 of a chemical or a solution in 5 c.c. of water of the residue remaining after any special treatment. Acidification is not necessary in the case of inorganic acids. Then add 10 c.c. of a saturated solution of  $\text{SO}_2$ , heat in a small beaker on a water-bath until all  $\text{SO}_2$  is driven off and the volume is about 2 c.c. Dilute this to about 5 c.c. with water.

*Test Apparatus.*—Prepare several generators as described below. Select a bottle of about 50 c.c. capacity, having a mouth of 2·5 cm. diameter ; fit with a suitably perforated rubber stopper. Insert in one perforation, a thistle funnel reaching to within 2 mm. of the bottom of the bottle. In the other aperture insert a vertical tube about 13 cm. total length and 1 cm. diameter for about 10 cm. but constricted at the lower extremity, to 5 mm. in diameter for 3 cm. of its length. This drawn-out portion should extend only slightly below the stopper. In the lower part of this exit-tube is to be

inserted a small pledget of dry glass wool and then a strip of the freshly-prepared but dry lead acetate test paper rolled into a coil, and above this a plug of the moist (not wet) lead acetate glass wool. In the upper extremity of this tube insert through a perforated cork stopper, a glass tube 12 cm. in length, having an internal diameter of about 3 mm. In this is to be placed the  $\text{HgBr}_2$  test paper, bending or creasing the upper portion of the strip so that it will retain its position. The strip should extend within about 2 cm. of the perforated cork stopper and must not be introduced into the tube until ready to start the test. This tube should be thoroughly cleaned and dried each time it is used.

*Preparation of Standard Stain.*—Introduce into the generator about 8 Gm. of the Zn followed by 25 c.c. of dilute  $\text{H}_2\text{SO}_4$  (1 in 4) and 5 drops of the acid  $\text{SnCl}_2$  reagent. Insert the stopper containing the thistle tube and the exit tubes into which have been placed the glass wool pledget, the dry lead acetate test paper, the moist lead acetate glass wool, and the  $\text{HgBr}_2$  test paper as described under the Test Apparatus. Add at once through the thistle tube 2 c.c. (accurately measured) of the standard  $\text{As}_2\text{O}_3$  solution and wash this down into the apparatus with 5 c.c. of the dilute  $\text{H}_2\text{SO}_4$  (1 in 4). Should the evolution of the gas be violent at first, check the reaction by immersing the bottle in cold water. Should the reaction subside, increase it by placing the bottle in warm water. If the reaction be too violent, the stain will spread and not form a distinctive band, thus making the colour intensity comparisons difficult. After the test has continued for 45 minutes, remove the mercuric bromide test paper and place it in a clean, dry tube for comparison. This stain represents 0.002 Mgm. of  $\text{As}_2\text{O}_3$  in addition to any stain produced by the reagents. The stain from the reagents should scarcely be perceptible when determined by a blank experiment. For preservation, the standard test strips are to be dipped into hot melted paraffin.

*Testing the Chemical.*—Repeat the above process, using the solution described under "Preparation of the Chemical" instead of the standard  $\text{As}_2\text{O}_3$  solution. The stain produced by the chemicals tested should not exceed in length or intensity of colour that prepared as a standard, indicating not more than 1 part of arsenic in 100,000 parts of the substance tested.

*Antimony*, if present in the substance tested, will produce a grey stain. Sulphites, sulphides, thiosulphates, and other compounds which liberate  $\text{H}_2\text{S}$  or  $\text{SO}_2$ , when treated with  $\text{H}_2\text{SO}_4$ ,

must be oxidized by means of  $\text{HNO}_3$  and then reduced by means of  $\text{SO}_2$  as directed under "Preparation of the Chemical," before introducing into the apparatus.

**REAGENTS.**—*Arsenious Acid*, Pure  $\text{As}_2\text{O}_3$ . *Pure Filter Paper*.

*Pure Glass Wool*.—Two Gm. digested on water-bath with 100 c.c. of dilute  $\text{HCl}$  filtered, evaporated and dried at  $110^\circ\text{C}$ . should not leave more than 0.01 Gm. of residue. 1 Gm. of glass wool boiled in a mixture 25 c.c. of dilute  $\text{HNO}_3$  and 25 c.c. of water; filter, evaporate to dryness, treat residue with 10 c.c. of water and again filter. The filtrate should not be affected by  $\text{H}_2\text{S}$  solution (lead).

*Lead Acetate Glass Wool*.—Immerse glass wool in a mixture of equal parts of lead acetate reagent and water, drain, dry on glass at  $100^\circ\text{C}$ .

*Lead Acetate Reagent*.—A 1 : 10 solution of clear crystals of  $\text{Pb } 2\text{C}_2\text{H}_3\text{O}_2 \cdot 3\text{H}_2\text{O}$ .

*Mercuric Bromide Test Paper*.—Cut stiff, heavy quantitative filter-paper into strips 3 cm. in width and 12 cm. in length. Immerse these strips for 5 minutes in alcoholic  $\text{HgBr}_2$  reagent. Remove the excess of solution by pressing the strips between filter-paper and then dry them quickly on glass in an oven heated to  $100^\circ\text{C}$ . Place the strips at once in a wide-mouthed bottle and stopper it securely.

*Mercuric Bromide Test Solution, Alcoholic*.—Dissolve 5 Gm. of  $\text{HgBr}_2$  in 100 c.c. of alcohol, employing a gentle heat to facilitate solution. Keep it in glass-stoppered bottles protected from the light.

*Stannous Chloride*,  $\text{SnCl}_2 + 2\text{H}_2\text{O}$ .—Colourless crystals readily soluble in water and alcohol. When in contact with air or excess of water, the salt readily forms a basic chloride, hence when dissolved, its solutions should be acidified with hydrochloric acid. The presence of arsenic above the U.S.P. limits should be determined. Boil 2 Gm. of the salt with 10 c.c. of  $\text{HCl}$  for several minutes; the solution should remain clear and colourless for one hour. When tested for As as directed under the blank test for As, 0.3 Gm. of  $\text{SnCl}_2$  should not produce a stain. *Alternative process*: Heat in with concentrated hydrochloric acid, taking care that the metal is in excess. When the acid has become saturated, pour off the clear fluid from the undissolved excess of tin, filter it through asbestos and set it aside to crystallize. Break up the crystals, drain, and dissolve them at once as directed under the Test Solutions or in Betten-



dorf's Arsenic Test. When thus prepared,  $\text{SnCl}_2$  must respond to the tests for arsenic given above. When freshly prepared the salt should be completely soluble in one part of alcohol (foreign salts).

*Stannous Chloride Test Solution, Acid.*—Dissolve 40 Gm. of  $\text{SnCl}_2$  crystals in 60 c.c. of concentrated  $\text{HCl}$  and preserve it in a glass-stoppered bottle.

*Sulphuric Acid, Concentrated for Tests,  $\text{H}_2\text{SO}_4$ .*—When concentrated sulphuric acid is especially directed in a test, it is intended that the strongest pure acid of a sp.g. of not less than 1.834 at  $25^\circ\text{C}$ . be employed. In addition to the tests prescribed for this acid in the text of the Pharmacopœia, it is required to conform to the following more rigorous tests before it can be employed as a reagent. Dilute 1 part of the acid with 4 parts of distilled water; the stain from 25 c.c. of this dilute acid should scarcely be perceptible when subjected to the arsenic test. Pour 1 c.c. of diphenylamine carefully so as to form a separated layer upon 5 c.c. of the concentrated  $\text{H}_2\text{SO}_4$  contained in a test-tube; no distinct blue colour should appear at the zone of contact (nitric acid). Upon carefully pouring about 2 c.c. of  $\text{HCl}$ , in which a particle of  $\text{Na}_2\text{SO}_3$  has been dissolved, over about 2 c.c. of the concentrated  $\text{H}_2\text{SO}_4$ , no reddish zone should appear and no precipitate should form (selenium).

*Tin, Sn.*—Pure metallic tin in the granulated or mossy condition. Digest 5 Gm. of tin with  $\text{HNO}_3$  on a water-bath until entirely converted into a white powder, then evaporate it completely to dryness. Stir the residue with 25 c.c. of diluted  $\text{HNO}_3$  and 25 c.c. of distilled water and filter it. To the filtrate add 1 c.c. of diluted  $\text{H}_2\text{SO}_4$ , evaporate it as far as possible upon a water-bath, and to this add 10 c.c. of distilled water; no weighable residue should remain undissolved (lead). When converted into  $\text{SnCl}_2$  it should comply with the tests directed under that salt.

*Zinc for Arsenic Test.*—The Zn should preferably be in globular form, about 3 to 6 mm. in diameter, known as No. 7 Shot Zinc. It should be free from S and P. The stain from 8 Gm. of Zn should scarcely be perceptible when determined by a blank experiment.

*Heavy Metals Test.*—Acidify 10 c.c. of a solution of the substance in distilled water (1 in 50) contained in a test-tube of about 40 c.c. capacity with 1 c.c. of diluted  $\text{HCl}$  (unless otherwise directed), warm it to about  $50^\circ\text{C}$ ., add an equal volume of freshly

prepared  $H_2S$  reagent, and allow the mixture to stand in a well-stoppered test-tube, in a warm place, at  $35^\circ C.$  for half an hour. At the end of this time the mixture should still possess the odour of  $H_2S$ ; if not, it should be thoroughly saturated with the gas and again set aside for half an hour. Any change in the colour of the solution which is being tested should be noted by comparison with the same volume of the  $H_2S$  reagent (which has been likewise acidified) when viewed crosswise by reflected light while held against a white surface.

**Viburnum opulus Bark wrongly described in U.S.P.** (*Drugg. Circ.*, 1914, 58, 652.) It is stated that during the past 10 years the bark of the maple *Acer spicata* has been substituted for genuine cramp bark from *Viburnum opulus*, and that the official U.S.P. monograph describes the substitute rather than the genuine drug. Consequently the drug is to be deleted from the next U.S.P., but finds a place in the N.F. where it is correctly described.

**Zinc Ointment, Needed Change in Formula for.** E. R. J o n e s. (*J. Amer. Pharm. Assoc.*, 1915, 4, 283.) Zinc ointment made with the official basis, benzoated lard (similar to the B.P. but containing 20 per cent. of  $ZnO$ ), goes granular on keeping. It is suggested that white petrolatum should be substituted for benzoated lard, and since the ointment is somewhat soft, hardened by the addition of wax, as follows: Zinc oxide, 200 Gm.; white wax, 150 Gm.; white petrolatum, 650 Gm. Rub the zinc oxide, which must be free from gritty particles, with an equal weight of melted white petrolatum until smooth and add to this the remainder of the white petrolatum which has been previously melted with the white wax. Strain the ointment while warm and stir thoroughly until it congeals. The strength of 20 per cent. is considered to be unnecessary. A 10 per cent. ointment as the B.P. is sufficiently strong.

## NOTES AND FORMULÆ.

**Almond Cream.** P. C a u l d w e l l. (*Drugg. Circ.*, 1915, 59, 373.) White wax, 8 oz.; spermaceti, 8 oz.; expressed oil of almond, 8 oz.; borax, 2 oz.; soap, 16 oz.; flake tragacanth, 2 oz.; oil of bitter almond, 2 drachms; oil of bergamot, 1 drachm; water, enough to make 1 gallon 4 pints 16 fl. oz. Melt the wax and spermaceti and add the expressed oil of almond.

Dissolve the borax in 64 fl. oz. of water and after warming to the same temperature as the melted wax and spermaceti, mix the two fluids, stirring just enough to ensure thorough admixture, and no more. Dissolve the soap in 6 pints 8 fl. oz. of water, made hot, and add to the mixture, being careful not to stir enough to make an undesirable foam. The tragacanth and 3 pints 8 fl. oz. of water must previously have been made into a smooth mucilage. Add this, brought to the same temperature, to the soap mixture, stirring briskly. Finally add the essential oils, and strain.

**Analgesic Balm.** R. FULTON. (*Midland Drugg.*, 1915, 49, 197.) Methyl salicylate, 2 oz.; menthol,  $\frac{3}{4}$  oz.; (or oil of peppermint,  $1\frac{1}{2}$  oz.); paraffin, 4 oz.; anhydrous wool-fat, 4 oz.; petrolatum, 4 oz. Put up in 1 oz. jars and label: An external application for the immediate relief of pain in almost any part of the body. Apply freely and cover with flannel. (Not to be applied to very tender surfaces.)

**Artificial Teeth, Powders for Cleaning.** (*Nat. Drugg.*, 1914, 44, 504.) (1) Prepared chalk, 42 oz.; powdered soap,  $\frac{1}{2}$  oz.; powdered borax,  $\frac{1}{2}$  oz.; oil of wintergreen, 10 minims. (2) Powdered cuttlefish bone, 2 oz.; precipitated chalk, 2 oz.; sodium bicarbonate, 1 oz.; oil of lavender, 10 minims. (3) Prepared chalk, 42 oz.; light magnesium carbonate, 8 oz.; powdered cuttlefish bone, 8 oz.; powdered pumice, 4 oz.; oil of peppermint, 5 minims.

**Ashes of Hedge Clippings and Trimmings as a Source of Potash.** E. J. RUSSELL. (*J. Board of Agric.*, 1914, 21, 694.) The ash of bonfires composed of threshing-waste, grass, weeds, dead and green wood, etc., was found to contain 11 per cent. of  $K_2O$ , a percentage nearly equal to that of kainite, which contains about 12.5 per cent. of  $K_2O$ . Thus some of the usual processes of farm routine give rise to ash rich in  $K_2O$ , which would in normal times be worth about 40s. a ton, and is now worth much more. Since the K is present in the ash as  $K_2CO_3$ , and therefore soluble in water, care must be taken to prevent loss due to rain, as experiments showed that a single night's rain of less than 0.1 inch diminished the  $K_2O$  in a heap of ash by 50 per cent. The cleaning out of hedge bottoms yielded about 5 lb. and hedge-trimmings on the average 15 lb. of ash per 100 yards of hedge (one side only). A 20-acre field with 1,300 yards of hedge

would, therefore, on this basis yield ash equivalent to more than  $\frac{1}{2}$  cwt. of kainite from the hedge bottoms, and to nearly 2 cwt. from the total trimmings. It was found that the cost of labour for obtaining and burning such clippings worked out at from 3*d.* to 8*d.* per lb. of ash when the material was mainly grass, and about 2*d.* a lb. when more wood than grass is present. Even when the ash could be obtained for 1*d.* a lb.  $K_2O$  obtained in this form would be very expensive if charged with the whole cost of the process, but where the trimming, etc., has to be done in any case, would well repay the trouble of collection.

**Book-keeping for Chemists.** A. H. Hills. (*Pharm. J.*, 1915 [4], 40, 556.) A valuable and lucid paper on this vitally important phase of business, specially adapted to the requirements of the retail pharmacist.

**Boroglycerin-Lanolin.** F. Wipperf. (*Pharm. Zeit.*; *Nat. Drugg.*, 1914, 44, 294.) The following formula for a cosmetic cream yields a preparation which, when rubbed upon the skin in moderate quantity, will not produce a fatty surface, a decided advantage when it is to be used upon the hands and face: Crystallized boric acid, 3; distilled water, 36; glycerin, 36; lanolin, hydrous, 30; white petrolatum, 195. The boric acid is dissolved in the mixture of glycerin and water with the aid of the heat of a water-bath. The white petrolatum is melted and the lanolin incorporated with it by stirring. The boric acid solution is then added, and the entire mixture thoroughly mixed until cold.

**Cacao or Chocolate Specialities.** (*Pharm. Zeit.*; *Schwiz. Apoth. Zeit.*, 1914, 52, 722.) The following formulæ are suitable for special "cocoas" or chocolates for invalids or dietetic purposes. The cacao used should contain about 20 per cent. of cacao butter. *Oatmeal cocoa.*—Cacao powder, 1,000; powdered roasted oatmeal, 1,000; vanillin, 0.5. *Nutritive cocoa.*—Cacao powder, 1,000; salt, 4; potassium phosphate, 20; calcium hypophosphite, 10; magnesium sulphate, 20; magnesia, 5. *Nutritive chocolate powder.*—Cacao powder, 450; maize meal, or rice powder, 50; sugar, 500; with the above salts, except the  $MgO$ . *Milk cocoa.*—Dried milk, 1 kilo; cacao powder, 1 kilo;  $MgO$ , 5 Gm. *Powdered milk chocolate.*—Cacao powder, 300; dried milk, 300; powdered sugar, 400; vanillin, 0.3;  $MgO$ , 5.

*Powdered cream chocolate* is similar, substituting cream powder for milk powder.

**Casein Massage Creams.** H. C. Bradford. (*Drugg. Circ.*, 1915, 59, 291.) Typical and original formulæ for these greaseless creams are given. (No. 1) Skimmed sweet milk, 1 gal. 3 pts. 4 fl. oz. ; solution of formaldehyde, 2 drachms ; borax,  $3\frac{1}{4}$  oz. ; alum,  $7\frac{1}{2}$  oz. ; boiling water, 3 pts. 4 fl. oz. ; cold water, 1 gal. 4 pts. 16 fl. oz. Mix the formaldehyde solution thoroughly with the milk and heat the solution to 50°C. Any desired colour should also be added to the milk at this time. It is thus carried down with the curd and distributed in a much better manner than is otherwise possible. One hundred and thirty-five minims of the solution of carmine, N.F., has been found to give a satisfactory tint to the above quantity. Now dissolve the borax in 32 fl. oz. of boiling water, and stir briskly into the milk ; as soon as the mixing is complete, strain it through muslin or cheese cloth. Dissolve the alum in the remainder of the boiling water and add the solution slowly and with constant stirring to the milk mixture. Let the curd settle to the bottom of the vessel, and if the supernatant liquid is not perfectly clear, add more alum solution of the strength stated above until it is. This done, drain off the liquid and wash the curd until the washings are tasteless or nearly so. Now get the curd into a bag of cheese cloth and press it with the hands, and let drain until it weighs 3 lb. and 2 oz. This will give about the proper amount of water. Next work in the perfume, and it is ready for package. If the process has been done properly, and if the cream has not been allowed to dry too much while getting down to the proper weight, it will be found satisfactorily smooth. However, if the air has gained too much access, it will be more or less "grainy," especially around the edges. If this graininess is only slight it will be best to simply remove and discard the affected portion, but if it is general, it had best be treated with a trace of caustic alkali. Either KOH or NaOH may be used, or a mixture of the two. Ten grains to the ounce of product is about the maximum, and probably less will serve. It all depends on the extent of the drying process. Dissolve the alkali in a little water—the least that will serve, and rub it smoothly and evenly into the curd. It will smooth out and dispel rough granular spots almost like magic. As soon as this is seen to be done, get the product into the tubes with the least possible delay. Tubes are the best

package in some respects, and in some others they are almost the very worst. Small, wide-mouthed bottles with ground stoppers are also used. (No. 2) Glycerin, 1 oz. ; ammonia water, 1 oz. ; borax, 2 drachms ; boric acid, 1 drachm ; fresh skimmed milk, 6 pts. 8 fl. oz. Mix the milk and the ammonia, then put on the fire and heat until the milk curdles. Let it stand over night or about 12 hours, and strain through cheese cloth. If it was heated sufficiently, and not too much, this will give a nice, smooth curd. Let it stand another 12 hours, then mix in the other ingredients, add the colour and perfume, and it is ready for packing. (No. 3) Freshly precipitated casein, 100 parts ; boric acid, 20 parts ; oil of theobroma, 10 parts ; colour ; perfume, of each, a sufficient quantity. Melt the oil of theobroma and rub to a paste with the boric acid, and triturate to a smooth, even cream with the casein, working in the perfume and colour at the same time. The casein is precipitated as follows : Fresh skimmed milk, 500 parts ; magnesium sulphate, 50 parts ; alum, 5 parts. Dissolve the  $MgSO_4$  in just the amount of warm water that will serve ; mix the solution with the milk and set the mixture aside for an hour or so. Heat it then to about  $130^{\circ}F.$  (and in no case allow the temperature to exceed  $145^{\circ}F.$ ), and add the alum, dissolved also in just sufficient hot water. Continue the heat until it is clear that the casein is entirely precipitated, then transfer to a cheese-cloth strainer, and wash with water until the washings are almost tasteless. It is then handled as directed above. This formula brings in a new element in the shape of a fat. This could be, and often is, added to the product of any formula, but its only use here is to exemplify the use of a particular fat. Cocoa butter seems to be by far the most popular for this purpose, since it imparts smoothness to the product. This lack of smoothness is the chief defect of most massage creams. The only disadvantage of this added fat is that the product is no longer, in the strict sense of the term, a "greaseless" cream. (No. 4) Fresh skimmed milk, 6 pts. 8 fl. oz. ; borax, 3 oz. ; boric acid, 3 oz. ; powdered alum, 6 oz. ; glycerin,  $1\frac{1}{2}$  oz. ; sodium benzoate, 4 drachms. Put the borax and the boric acid into the milk, stir until dissolved, then heat just to the boiling point. Then remove from the fire, add the alum dissolved in the smallest possible quantity of hot water, stir thoroughly, and let stand for 24 hours. Strain, wash the curd a few times by decantation, let drain for an hour or so, then mix in the glycerin, the benzoate, the perfume, and the colour, and it is ready to

package up. (No. 5) *Dry casein cream*.—Dry commercial casein may be used, but care must be taken to secure a proper kind since some dry caseins are more suitable for making "size" than creams. With a suitable casein the following is satisfactory: Dry casein, 9 oz. ; KOH, 100 grains ; NaOH, 20 grains ; glycerin, 4 oz. ; phenol, 140 grains ; water, 32 fl. oz. Dissolve the alkalies in the water, add the casein, stirring until it is free from lumps. Heat on the water-bath until it forms a smooth heavy creamy mass. Incorporate the glycerin and other ingredients. This formula enables a cream of any desired consistence to be produced ; firmer than the above by lessening the amount of water ; or thinner, by increasing it. *Curd cheese as massage cream basis*.—The ordinary curd cheese forms one of the best bases for massage cream, after the addition of 20 minims of phenol to the pound and suitably colouring and perfuming, rubbing to a smooth paste in a mortar.

**Castilian Tooth Wash.** (*Nat. Drugg.*, 1915, 43, 23.) White Castile soap, 3 oz. ; glycerin, 5 fl. oz. ; filtered water, 20 fl. oz. ; alcohol, 30 fl. oz. ; oil of peppermint, 1 fl. drachm ; oil of wintergreen, 1 fl. drachm ; oil of orange peel, 1 fl. drachm ; oil of anise, 1 fl. drachm ; oil of cassia, 1 fl. drachm. Beat up the soap with the glycerin in a mortar ; dissolve the oils in the alcohol, and pour upon the soap and glycerin contained in a bottle. Shake well until the soap is completely dissolved. Then colour to suit with a solution of carmine.

**Chemical Manure for Gardens.** (*J. Board Agric.*, 1914, 20, 978.) Commercial ammonium sulphate (95 per cent.), 2 ; superphosphate of lime (26 per cent. soluble phosphate), 12 ; bone meal, finely ground, 3 ; commercial potassium sulphate (90 per cent.), 3. Mix and sift through coarse sieve. Store in a dry place. Apply in quantities not exceeding 1 lb. to 4 square yards.

**Cockroach Poison, Sodium Fluoride as.** — **M e a n s.** (*Naval Medical Bulletin ; Drugg. Circ.*, 1915, 59, 168.) Cockroaches and other insect pests were destroyed in the store-rooms and pantries of a ship of the U.S. Navy by scattering NaF as an insecticide.

**Cold Cream and Hand Lotions, Discussion on.** (*J. Amer. Pharm. Assoc.*, 1915, 4, 158.) Gray is of opinion that the essentials of a formula for cold cream depend upon whether it is desired to

be absorbent or non-absorbent. If it is to be absorbed a vegetable oil must be used, but if not, mineral oil. Two formulæ are given for superior products. *Absorbent Cream*.—Oil of peach-kernels, 8 oz. ; white wax, 1 troy oz. ; spermaceti, 1 troy oz. ; water  $2\frac{3}{4}$  oz. ; borax, 5 grains ; oil of rose, 25 minims ; oil of patchouli, 1 minim. Dissolve the wax in the oil, use gentle heat or preferably a water-bath. When the wax is dissolved, add the water, previously heated, and in which the borax has been dissolved, slowly, constantly stirring with an egg-beater. When cold, add the perfume oils. Non-absorbent, but nicer in appearance, is the product produced by using the following formula : *Theatrical Cream*.—White Russian paraffin oil, 8 oz. ; ceresin and white wax of each, 2 troy oz. ; water,  $3\frac{1}{4}$  oz. ; borax, 5 grains ; oil of rose, 25 minims ; oil of patchouli, 1 minim. Proceed as in the former formula. Any cheaper odour may be used, such as Almond, orange flower or synthetic rose. Hand lotions should have emollient properties, be thick yet easily absorbed or dried on the hands, with a pleasing odour and appearance. A formula which has given satisfaction is : Powdered tragacanth,  $1\frac{1}{2}$  troy oz. ; alcohol, 16 oz. ; glycerin, 8 oz. ; benzaldehyde, 60 minims ; oil of lavender, 20 minims. Mix thoroughly, and add quickly water enough to make one gallon. Lascoff stated that the following formula is satisfactory : *Cold Cream*.—White wax,  $12\frac{1}{2}$  oz. ; liquid paraffin, 48 oz. ; distilled water, 24 oz. ; borax, 6 drachms ; oil of rose, q.s. For dispensing purposes however the formula of the Pharmacopœia should be used and nothing else. Raubenheimer stated that the official formula for Ointment of Rose Water can be modified into a Theatrical Cold Cream by using paraffin oil in place of the oil of almonds. The cold cream produced by use of this process has the great advantage of keeping perfectly without change. There is consequently a large demand for such a preparation.

**Cold Cream, Chemistry of.** H. S. Groat. (*J. Amer. Pharm. Assoc.*, 1915, 4, 169.) The paper is mainly theoretical. A number of equations are given to show the reaction supposed to occur between borax and glycerol and the various glycerides used in cold cream. The following formula is stated to give a satisfactory preparation : Oil of sweet almonds, 77 ; wool fat, 15 ; paraffin lard, 18 ; white wax, 18 ; borax, 1.5 ; hydrogen peroxide, 1.5 ; oil of rose geranium, 0.2 ; distilled water, 27 ; otto of rose, 0.4.



**Cold Cream, Greasy.** E. R. Jones. (*J. Amer. Pharm. Assoc.*, 1915, 4, 708.) The author does not agree with the theoretical statements of Groat as to the chemical changes which occur in making cold creams. These preparations are essentially emulsions and require a soap or some other emulsifying agent to be present. Borax is considered to be the best saponifying agent, combining with the free cerotic acid of the wax, the soap acting as an emulsifying agent. White beeswax, paraffin or ceresin, colourless liquid petrolatum (mineral oil), borax and water in proper proportions form the best creams. Creams made from these ingredients are more permanent than when vegetable oils are used and do not turn rancid. Much attention should be paid to obtaining tight-fitting covers to guard against loss of water.

**Condition Powders, American.** (*Nat. Drugg.*, 1914, 44, 506 ; Bloodroot, 1 lb. ; capsicum, 1 lb. ; sassafras, 2 lb. ; copperas, 2 lb. ; ginger, 3 lb. ; gentian, 3 lb. ; black sulphide of antimony, 3 lb. ; saltpeter, 4 lb. ; resin, 10 lb. ; fenugreek, 10 lb. ; salt, 20 lb. ; sulphur, 25 lb. ; linseed, 25 lb. All of the drugs except the linseed should be in fine powder ; for the last named, ordinary ground linseed may be used. Thoroughly mix and put in pound cartons lined with waxed paper.

**Copper Sulphate to Disinfect Water in Swimming Baths.** S. J. Thomas. (*J. Ind. Eng. Chem.*, 1915, 7, 496.)  $\text{CuSO}_4$  in the proportion 0.04 parts per million of water is an efficient bactericide for use in swimming baths, preferable to chlorinated lime. It is more effective because it does not undergo chemical change readily. Chlorinated lime owes its power to the chemical change and is afterwards useless. It is not irritating to the eyes and mucous membranes as is "hypochlorite" if the latter is used in germicidal quantities ; it is cheaper and has no odour. If all other conditions were equal this last fact alone would prove its great advantage over chlorinated lime. Copper sulphate when used in water in quantities enormously in excess of what is necessary to destroy all pathogenic germs is quite harmless to the bathers.

**Corn Paint.** Gaucher. (*Practitioner ; Pharm. J.*, 1915, 40, 29.) Resorcin, 15 grains ; salicylic acid, 15 grains ; lactic acid,  $2\frac{1}{2}$  fl. drs. ; flexible collodion,  $2\frac{1}{2}$  fl. drs. This is to be applied for five or six days in succession. The foot is then well

soaked in hot water ; the film on being lifted off brings the corn away with it. The author claims that this treatment will remove the most inveterate corn.

**Digestive Tablets.** (*Nat. Drugg.*, 1915, 45, 23.) Diastase, 25 gr. ; heavy magnesium carbonate, 500 gr. ; pancreatin, 25 gr. ; pepsin, 100 gr. ; precipitated chalk, 100 gr. ; refined sugar, in powder, 800 gr. ; milk sugar, 500 gr. ; oil of cinnamon, a sufficient quantity ; oil of coriander, a sufficient quantity. Mix and divide into 100 tablets.

**Dispensing Counter and Laboratory Bench Tops, Stain and Finish for.** F. W. Nitardy. (*J. Amer. Pharm. Assoc.*, 1914, 3, 967.) If previously varnished, thoroughly remove the varnish by planing or otherwise. Thoroughly clean the wood by scrubbing with soap and water. Allow it to dry. Prepare a saturated solution of  $\text{KClO}_3$ , heat to boiling, and apply to the wood while hot, so that it will penetrate the fibre. When dry, apply a second coat, in the same manner. Now prepare a 20 per cent. solution of  $\text{CuSO}_4$  and apply boiling hot, after the former has dried, allowing the wood to become well saturated and taking up any surplus liquid remaining after 10 to 15 minutes, so that no appreciable crystallization takes place on top of the wood. When this is dry, apply a solution made by dissolving 90 parts by volume of aniline oil in 60 parts by volume of  $\text{HCl}$ , diluted to 500 parts with water, and allow that to well penetrate the wood. Let this coat dry about 6 hours or over night, then apply a heavy coat of hot, raw linseed oil. Allow to stand 6 hours or over night and scrub well with soap and water until all surplus colour has been removed, that is until the water stays clean, now allow to dry and rub down well with linseed oil, applying several coats (a day or two apart) if necessary to completely fill the pores of the wood. This gives a deep-black finish, with a slight gloss, which can be kept in perfect condition by an occasional scrubbing with soap and water and a subsequent rub-down with linseed oil.

**Drying Oven Temperatures, Want of Uniformity in Gas and Electrically Heated Ovens.** L. H. Bailey. In small hot water ovens  $15 \times 8$  inches a uniform temperature of  $100^\circ\text{C}$ . is easily attained. In a larger oven  $15 \times 15 \times 15$  inches the maximum variation may be  $4^\circ\text{C}$ . or on one shelf  $3^\circ\text{C}$ . The best electrically heated ovens maintained approximately at  $100^\circ$  showed a

variation of 10°C. on the upper shelf, 13°C. on the lower shelf, and 15°C. between the coolest spot on the lower shelf and the hottest on the upper. Gas-heated air ovens show about the same variations as electrically heated ones.

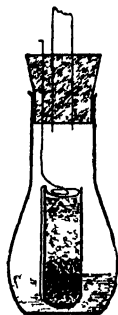
**Dusting Powder to Destroy Vermin.** Langford Moore. (*Lancet*, 1915, 188, 529.) The author recommends the local application of a mixture of ammoniated mercury, 2; zinc oxide, 1; French chalk, 1. This is applied freely to the infested region. No toxic effects are produced, and no mercury is to be found in the urine after the use of the powder. When it has been applied to infested suppurating wounds, both parasites and their ova have been destroyed in two days.

**Emulsification, Theory of.** W. D. Bancroft. (*J. Phys. Chem.*, 1915, 19, 275; *J.S.C.I.*, 1915, 34, 560.) Hydroxyl ions can be absorbed from an alkaline aqueous solution by many organic liquids. When solid particles are shaken with water and an immiscible organic liquid, the phase into which they will tend to pass will depend upon which liquid they absorb. If they absorb both liquids it is probable that a homogeneous liquid phase is formed about their surfaces. The space between the two liquids into which a suspended substance wetted by them both will pass, is termed the "dineric interface." If the particles are unable to coalesce into a coherent film the emulsion that tends to form will not last long (e.g. copper powder, kerosene, and water). The name "interfacial" is given to substances that pass into the dineric interface of two liquids, and if suspended in one liquid they can be shaken from their suspension by the addition of a second liquid towards which they are interfacial. Winkelblech's test for colloids is a means of detecting interfacial substances, and is applicable when a fairly stable emulsion is formed or when the interfacial substance is not readily brought into colloidal solution by either liquid. When an interfacial substance is withdrawn from an aqueous liquid it is probably less hydrous in proportion to the amount of surface of the other liquid. Experiments tend to confirm the view of Briggs that Winkelblech's method will detect substances in colloidal solution in the presence of dissolved substances. In the author's experiments with the method,  $C_6H_6$  gave a faint film in the absence of gelatin; this was found to be due to the presence of traces of resins, and the test may therefore be used for the detection of such impurities in  $C_6H_6$ .

**Emulsions and Emulsification.** F. G. D o n n a n. (*J.S.C.I.*, 1915, 34, 560.) In a lecture before the Royal Institution the factors determining the formation, stability, and destruction of emulsions were dealt with. Emulsions of water in oil and of oil in water were projected on the screen and their structure and the motion of the emulsion particles in an electric field was shown. The lecturer explained how the stability depended on the particles carrying electric charges, and showed how the variation of the electric charge, as dependent on the addition of acids, alkalies, and salts, affected the stability of the emulsion. Experiments were shown illustrating the effect of electric discharges in coagulating and settling dusts, fumes, and emulsions. In the second part of the lecture the part played by surface tension and surface-concentration (surface absorption) was discussed. Experiments were shown illustrating the effect of various emulsifying agents on surface tension, and their power in producing stable emulsions. The lecturer explained how stability was caused by the production of concentrated surface layers or surface skins. Experiments were shown demonstrating the formation of such concentrated surface-layers, both at water/-air and water/mercury surfaces. The stability of the fat emulsion in milk was dealt with, and a sample of synthetic artificial milk was exhibited. In conclusion, the lecturer pointed out that the two main factors determining the stability of emulsions, namely electric charge and surface layer, were also to be met with in dealing with colloidal solutions. The substances which lowered the surface tension and therefore formed concentrated surface layers (emulsifying agents) corresponded in some degree to the "protective" colloids, which play such an important part in the technical and medical applications of colloidal solutions. Reference was also made to the possibility that the formation of surface layers may be in part due to electrical absorption.

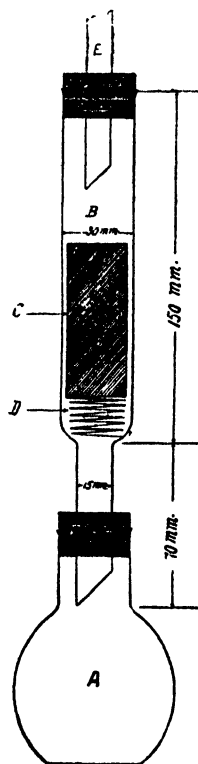
**Extractor, Simple.** G. A. S t o k e s. (*Analyst*, 1914, 39, 295.) The apparatus consists of a flask, a piece of wire, and a thimble of fat-free paper. Through the cork connecting the flask with the condenser passes a wire, so bent as to hold a fat-free paper thimble within the flask. Into this thimble the substance to be extracted is placed, and above it a layer of fat-free cotton-wool to prevent any of the substance floating over. The wire is then bent into a spring so as to allow the thimble to be inserted and the cotton wool pressed down. At the commencement of the

extraction the thimble and its contents are forced down by the wire so that they are immersed in the solvent. After boiling for a short time in the solvent, the hook of the wire on the outside is raised, without disconnecting any part of the apparatus, and the thimble brought out of the liquid, into the neck of the flask. As the space between the neck and thimble is narrow, the boiling vapour as it ascends must act upon the outside of the thimble. The thimble being pervious is constantly being washed inside and out by the hot solvent. When the extraction is finished, the flask is detached, the solvent evaporated, and the flask and its contents weighed.



**Extractor Tube, Simple.** C. A. Butt. (*J. Ind. Eng. Chem.*, 1915, 7, 130.) The following is claimed to be a

cheap and efficient substitute for the Soxhlet tube. The illustration shows the approximate dimensions. The vapour from the solvent passes from flask *A* through the small part of tube *B* and around thimble *C*, which is raised by the use of a wire coil, spring or gauze *D*, to allow free passage of vapour to the condenser tube *E*. The tubes are made of thick glass, and are, therefore, very durable.



**Fly Larvae and Pupae, to Kill.** (*U.S. Department of Agriculture ; Bulletin 118 ; Drugg. Circ.*, 1915, 59, 372.) As is well known, the house fly larvae live chiefly in stable manure and organic refuse. Borax is found to be the most efficient poison to destroy them. One pound of borax should be used for every 12 bushels (or 15 cubic feet) of manure. Special care should be taken to sprinkle some on the edges of the heap, where it comes in contact with the ground. This is where the larvae migrate to assume the pupal form. After treatment with borax 2 or 3 gallons of water should be sprinkled over the heap.

**Formalin as a Vermifuge for Dogs.** (*Pharm. J.*, 1915 [4], 40, 687.) A formalin tablet given twice or thrice daily for a week is an effective anthelmintic for dogs. It has succeeded in a case where calomel and santonin failed to effect a permanent cure. It does not occasion sickness.

**Hair-cleaning and Drying Powders.** (*Nat. Drugg.*, 1914, 44, 505.) (1) Orris root, white corn meal, equal parts of each in fine powder. (2) Orris root, 2 oz. ; French chalk, 3 oz. ; starch, 5 oz. ; tincture of musk, 20 minims ; oil of lemon, 15 minims ; oil of bergamot, 15 minims ; oil of neroli, 6 minims. (3) Wheat flour, 8 oz. ; orris root, powder, 1 oz. Heat the flour until perfectly dry, then mix intimately.

**Hand-cleaning Paste for Motorists.** (*Nat. Drugg.*, 1914, 44, 505.) Soft soap, 80.5 ; ammonia solution, 5 ; punice, finely powdered, 31 ; oil of turpentine, enough to form a paste. Fill into tubes.

**Hydrocyanic Acid in Horticulture.** F. P. Sargeant and F. C. Edwards. (*Gard. Chron.* ; *Pharm. J.*, 1914, 39, 193.) In order to obtain the HCN in the purest form for fumigating plant houses it is recommended that it should be generated from KCN 99 per cent., or NaCN "130 per cent." and  $H_3PO_4$ , sp.g. 1.5. Equal quantities of NaCN and acid are to be used. For green aphid: Sodium cyanide,  $\frac{1}{8}$  oz. ; phosphoric acid,  $\frac{1}{8}$  oz. ; water,  $\frac{1}{2}$  oz. for each 1,000 cubic ft. This will not scorch any plants. One application is sufficient. For black or white aphid, thrips and scale: Sodium cyanide,  $\frac{1}{4}$  oz. ; phosphoric acid,  $\frac{1}{4}$  oz. ; water, 1 oz. for each 1,000 cubic ft. This will not scorch mature plants. One application is sufficient. For mealy bug and red spider in house of mixed plants: Sodium cyanide, 1 oz. ; phosphoric acid, 1 oz. ; water, 4 oz. for each 1,000 cubic ft. Some few young shoots may be affected at this strength, but to a very slight extent only. For mealy bug on dormant vines, etc.: Sodium cyanide, 2 oz. ; sulphuric acid, 4 oz. ; water, 8 oz. for each 1,000 cubic ft. Fumigate three times at intervals of a week to ensure complete destruction. After vines have started growth it is unsafe to fumigate them. The best time is after the fruit has been gathered and before the leaves have fallen. At this period the mealy bug is active, and is more easily destroyed than later, when it reaches a semi-quiescent state. Experience has shown that 1 oz. of sodium

cyanide with phosphoric acid for each 1,000 cubic ft. is fatal to all ordinary pests, and that no injury ensues except to the young shoots of the most delicate plants. Even with twice this amount a large number of plants are unaffected, and injury is evident only to delicate or young organs of tender plants. In all cases the fumigation should be made in the evening when the plants are dry. The house should be closed up. Entry should not be made until the house has been opened for half an hour on the following morning.

**Immiscible Solvents, Extraction by Means of, from Point of View of Distribution Coefficients.** J. W. Marden. (*J. Ind. and Eng. Chem.*, 1914, 6, 315.) Several well-established "shaking-out" methods have been examined, with the result that some have been found to be less exact than was supposed, but to be readily capable of improvement, whilst others were found to be unnecessarily wasteful of solvents. For the estimation of acetanilide in  $H_2O_2$  solutions (*Bulletin* 150 of the U.S. Bureau of Chemistry) directions are given to shake the solution twice with  $Et_2O$  and once with  $CHCl_3$  and to evaporate the combined extracts, but no reason is given for the use of two solvents nor any suggestion made as to quantities. It is stated that the method extracts 95 per cent. of the acetanilide. This is the case if 20 c.c. portions of solvent are used to extract 50 c.c. of the solution, but if  $CHCl_3$  be used for each extraction 98.5 per cent. may be extracted. By using smaller portions of solvent at a time an equal volume may be made to give an even better result, or an equally good result may be obtained with economy of solvent.

For the extraction of saccharin from aqueous solutions (*Bulletin* 107 of the U.S. Bureau of Chemistry) directions are given to render the solution slightly acid with  $HCl$ , and to extract with  $Et_2O$  "as in the case of salicylic acid." The cases could hardly be less alike, viewed from the point of the respective distribution ratios. Salicylic acid may be almost quantitatively extracted with  $Et_2O$  by a few washings, whereas, from very faintly acid solutions, four washings with 20 c.c. portions of  $Et_2O$  will not extract as much as nine-tenths of the saccharin from 100 c.c. A better solvent was found in amyl alcohol, of which four 10 c.c. portions will extract 99.9 per cent. of saccharin from 50 c.c. of an acid aqueous solution. Amyl alcohol, however, being somewhat unpleasant to work

with, other methods were tried, and it was found that the  $\text{Et}_2\text{O}$  method was greatly improved by rendering the aqueous solution strongly acid (5 c.c. concentrated  $\text{HCl}$  per 100 c.c.). Under these conditions, four 20 c.c. portions of  $\text{Et}_2\text{O}$  were found to extract 99.5 per cent. of the saccharin from 100 c.c. of aqueous solution. In his method for the estimation of tannin in tea, Smith removes caffeine from 50 c.c. of aqueous extract by treatment with four successive portions of 30 c.c. of  $\text{CHCl}_3$ . It is shown that four portions of 10 c.c. would remove 99.7 per cent. of the caffeine.

**Infants' Foods, Proprietary, Analysis and Composition.** J. L. BAKER. (*Local Gov. Board Reports*, 1914, Food Report No. 20, 1; *J.S.C.I.*, 1914, 33, 882.) One hundred and six samples of different brands of proprietary infants' foods have been examined; certain of these were analyzed fully. The suitability of foods containing starch is discussed and a synopsis is given of the recorded evidence regarding the effect of such foods on infants. Misleading statements in the labels or advertisements of the foods are noted, and suggested methods for exercising control over the foods are given together with the regulations obtaining in other countries. Results of the analyses of 29 of the foods are given in detail. Four of the samples contained dried milk and all contained sucrose; usually the quantity of the latter was small, but in at least 8 cases the sugar must have been added, as the amount varied from 9.2 to 16 per cent. The fat-content was small in almost all the foods, not exceeding that naturally present in the flour used; the highest quantity of fat found was 16 per cent. All the samples, except two, contained appreciable quantities of unaltered starch; the majority contained over 60 per cent., the highest amount found being 75 per cent. Most of the foods were prepared from wheat flour, but oat, banana, and lentil starches were present in some of the samples. The proteins varied from 1.8 to 24.2 per cent., and the mineral matters from 0.5 to 3.8 per cent. Many of the foods contained saccharifying diastase, whilst liquefying diastase was present in about one-half of the samples.

**Insecticides, Horticultural Sprays and Disinfectants, Formulæ for.** E. V. HOWELL. (*J. Amer. Pharm. Assoc.*, 1915, 4, 312.) *Bordeaux Mixture*.—Copper sulphate, 560 grains; lime, 831 grains; water to make 6 pints 8 fl. oz. Dissolve the  $\text{CuSO}_4$ .



in half a gallon of water in one vessel, slake the CaO in another vessel and dilute with enough water to make half a gallon, pour them together simultaneously into a suitable vessel when a blue mixture should be formed. Used for apple leaf rust, apple scab, bitter rot of apple, black rot, downy mildew, leaf blight, anthracnose, wilt, powdery mildew, black spot canker, brown rot, fruit blotch, leaf curl, black knot, flyspeck.

*Bordeaux Mixture with Arsenic.*—Bordeaux mixture, 6 pints 8 oz. ; Paris green, 70 grains. Make the Paris green into a thick paste with water and add to the Bordeaux mixture previously prepared. Should be strained before being used. *Uses.*—Codling moth, common asparagus beetle, cucumber beetle, flea beetle, and many other insect pests.

*Bed Bug Killer.*—Camphor,  $12\frac{1}{2}$  oz. ; paraffin wax,  $12\frac{1}{2}$  oz. ; rape seed oil, 25 oz. ; benzin to make 1 gallon. Mix. *Kerosene Emulsion, with Whale Oil Soap.*—Kerosene, 85.3 oz. ; whale oil soap, 2.6 oz. ; water, 42.6 oz. Dissolve the soap in the water by the aid of heat, then immediately add the kerosene, make emulsion by churning. Used for various scale insects, larvae and aphides.

*Lime, Sulphur and Salt Wash.*—Unslaked lime, 6.4 oz. ; sulphur, 4.8 oz. ; salt, 3.2 oz. ; water to make 6 pints 8 oz. Slake the lime, add the sulphur and salt, and heat to boiling. Used as a general insecticide and fungicide for spraying trees before the leaves appear.

*Lice Exterminator.*—Naphthalin,  $3\frac{1}{2}$  oz. ; beeswax,  $1\frac{1}{2}$  oz. ; coconut oil, 5 oz. ; petrolatum,  $5\frac{3}{4}$  oz. ; bergamot oil,  $1\frac{1}{2}$  drachms ; clove oil,  $1\frac{1}{2}$  drachms ; cinnamon oil,  $1\frac{1}{2}$  drachms ; lemon oil, 50 minims. Melt the fats together, add the naphthalin and stir until the latter is dissolved, cool and add the oils.

*Mange Cure.*—Whale oil, 100 oz. ; sulphur, 6 oz. ; tar oil, 12 oz. ; any crude oil to make 6 pints 8 fl. oz.

*Naphthalin Solution.*—Naphthalin, 10 oz. ; lavender oil, 16 oz. ; alcohol 90 per cent., to make 6 pints 8 fl. oz. An application to keep away mosquitoes and other biting insects.

*Potassium Sulphide Spray.*—Potassium sulphide, 146 grains ; water to make 6 pints 6 fl. oz. Dissolve. For red spider.

*Adhesive Resin Wash.*—Powdered resin, 1 lb. ; concentrated lye, 3.2 oz. ; fish oil, 5 oz. ; water to make 6 pints 8 fl. oz. Place the oil and resin in 32 fl. oz. of water and boil until the resin is thoroughly softened. Dissolve the lye in a separate vessel and add it slowly to the resin mixture, stirring constantly until well mixed. Then add enough water to make the whole measure 6 pints 8 fl. oz. Continue the boil-

ing until the mixture will mix readily. *Uses*.—With a few plants like cabbage and collards which have very smooth foliage, difficulty is often experienced in making poison mixtures adhere and for this purpose this mixture is used. *Resin and Sulphur Solution*.—Sulphur,  $1\frac{1}{2}$  lb. ; powdered resin, 580 grains ; caustic soda, 1 lb. ; water to make 6 pints 8 fl. oz. Mix sulphur and resin and make into a thick paste with water. Dissolve the caustic soda in water and add to the first mixture, stir, after boiling ceases and the mixture has acquired a brownish colour add half a gallon of water and stir well, finally add water enough to make 6 pints 8 fl. oz. *Uses*.—Red scale, San Jose scale. *Solution of Mercuric Chloride*.—Mercuric chloride, 72 grains ; water to make 6 pints 8 fl. oz. Dissolve. Used to disinfect the knife or other tools in cutting out pear blight. *Ammoniated Copper Carbonate Solution*.—Stronger solution of ammonia, 1 oz. ; copper carbonate, 53 grains ; water to make 6 pints 8 fl. oz. Make the copper carbonate into a thin paste with water and slowly add the ammonia, then add enough water to make 6 pints 8 fl. oz. *Use*.—This insecticide is mainly used as a substitute for Bordeaux mixture upon ornamental plants and maturing fruits as it does not leave the stain that Bordeaux mixture leaves. It is also inferior as a fungicide. Destructive to powdery mildew. *Eau Celeste, Modified*.—Copper sulphate, 512.5 grains ; ammonia solution, 10 per cent., 3.2 oz. ; sal soda, 1.8 oz. ; water to make 6 pints 8 fl. oz. Dissolve the copper sulphate in 64 fl. oz. of water, add the ammonia and dilute with water to 6 pints 8 fl. oz. and dissolve in the sal soda. *Use*.—This wash should not be used on the foliage of stone fruits and should be applied to other growing plants only with due caution. *Soap Solution*.—Laundry soap, 6 oz. ; water to make 6 pints 8 fl. oz. Reduce the soap to fine shavings and dissolve by the aid of heat in half a gallon of water, then add enough water to make 6 pints 8 fl. oz. *Uses*.—Apple tree borer, fluted scale, melon plant louse, pear tree slug, red spider. *Solution of Larkspur and Bichloride of Mercury*.—Fluid extract of larkspur, 8 oz. ; bichloride mercury, 28 grains ; water to make 6 pints 8 fl. oz. Mix. Used against pediculi. *Solution for Itch*.—Lime, 1 lb. ; sulphur, 2 lb. ; water, 6 pints 8 fl. oz. Mix and boil for 1 hour, then strain.

**Myrrh Tooth Powder.** (*Nat. Drugg.*, 1914, 44, 504.) Powdered myrrh,  $2\frac{1}{2}$  drachms ; sodium chloride dried,  $2\frac{1}{2}$  drachms ;

powdered white Castile soap,  $1\frac{1}{2}$  drachms ; precipitated chalk, 16 oz. ; oil of rose, enough to flavour.

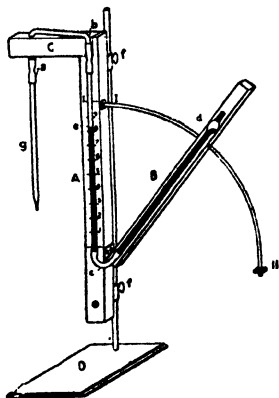
**Office Paste.** (*Pharm. J.*, 1914 [4], 39, 485.) Flour,  $2\frac{1}{2}$  oz. ; water, 13 oz. ; borax, 30 grains ; alum, 30 grains ; creosote, 5 minims ; oil of cloves, 5 minims. Cream the flour carefully with  $2\frac{1}{2}$  oz. of the water ; add the oils and  $4\frac{1}{2}$  oz. more of the water. In the remaining 6 oz. of the water, made hot, dissolve the borax and alum. Lastly, add the "cream," and cook thoroughly, but not so much as to effect loss by evaporation.

**Paste for Cleaning Glass.** (*Nat. Drugg.*, 1914, 44, 293.) Prepared chalk, 6 lb. ; powdered French chalk,  $1\frac{1}{2}$  lb. ; calcium phosphate,  $2\frac{1}{4}$  lb. ; powdered quillaia bark,  $2\frac{1}{4}$  lb. ; ammonium carbonate, 18 oz. ; rose pink, 6 oz. Mix the ingredients, in fine powder, and sift through muslin. Then mix with soft water to the consistence of cream, and apply to the glass by means of a soft rag or sponge ; allow it to dry on, wipe off with a cloth, and polish with chamois.

**Pastes for Tins.** (*Drugg. Circ.*, 1914, 59, 377.) The following are stated to be more satisfactory in practice than the formulæ usually published. *For Lacquered Tin.*—Wheat flour, 2 to 4 oz. ; corn starch, 2 drachms ; powdered alum, 2 drachms ; phenol (86.4 per cent.), 1 drachm ; clarified honey, 1 to 2 oz. ; balsam of fir, 1 oz. ; water, 8 oz. Mix the solids with the water and heat the mixture on a water-bath until a stiff paste results. Then add the phenol and the honey, and after mixing well slowly pour in the balsam of fir and stir until it is thoroughly distributed. In place of balsam of fir, 2 oz. of imitation Venice turpentine can be used. *For Unlacquered Tin.*—The following method is the best for unlacquered ware : Wipe off as much of the grease from the can as possible ; then apply to label a paste made as follows : Get 8 oz. of solution of sodium silicate, the heavy, thick and cloudy kind (the clear transparent sort is no good), and add  $\frac{1}{2}$  oz. of solution of KOH (1 in 10) and 1 oz. of glycerin. Mix these well together. The silicate may thicken when the glycerin is added, but with constant stirring it will thin out ; then if too thick, add enough boiling water to thin. One-half to 1 oz. is usually enough.

**Pipettometer.** W. D. Frost. (*J. Amer. Chem. Soc.*, 1914, 36, 1785-87.) Originally designed for bacteriological work this instrument may find useful application for other purposes.

It consists essentially of the straight glass tubes  $g$ ,  $e$ , and  $B$ , and the double right-angled bend  $b$ , connected by short pieces of rubber tubing as shown. The tube  $B$  has a bulb,  $d$ , near the top, and is supported on a wooden arm connected by a hinge to the wooden upright,  $A$ , to which  $e$  is fixed.  $A$  and the rest of the apparatus can be raised or lowered after loosening the set screws,  $ff$ . With  $d$  in a vertical position, mercury is poured in until it stands at some convenient high level, such as  $I$ , in the permanently vertical tube  $e$ . This point is marked. A beaker of water is brought under the point of the tube  $g$ , the arm  $B$  is lowered, the height of the mercury in  $e$  marked on the wooden backboard, and the contents of  $g$  discharged by raising  $B$  and weighed. By repeating this experiment with greater or less lowering of the arm  $B$ , data for the construction of the scale shown may be obtained. (Obviously a graduated pipette in place of  $e$  will not serve the purpose of a scale, since the air between the liquid in  $g$  and the mercury in  $e$  is under a variable pressure.



**Potassium Tellurite as an Indicator of Microbial Life.** W. E. King and L. Davis. (*Am. J. Pub. Health*, 1914, 4, 917-32; *Chem. Abstr. Amer. Chem. Soc.*, 1915, 9, 215.) Nearly all of the more common micro-organisms react with  $K_2TeO_3$ , forming characteristic, black compounds. This reaction depends on reduction. As a general microbic indicator a dilution of 1:50,000 is sufficient. This concentration produces no irritant action when introduced into test animals.

**Prescription Counter and Laboratory Table, Finish for.** F. W. Nitardy. (*Nat. Drugg.*, 1914, 44, 424.) Prepare a saturated solution of  $KClO_3$ , heat to boiling, and apply to the wood while hot, so that it will penetrate the fibre. When dry, apply a second coat in the same manner. Now prepare a 20 per cent. solution of  $CuSO_4$  and apply boiling hot, after the former has dried, allowing the wood to become well saturated and taking up any surplus liquid remaining after 10 to 15 minutes, so that no appreciable crystallization takes place on top of the

wood. When this is dry, apply a solution made by dissolving 90 parts, by volume, of aniline oil in 60 parts, by volume, of HCl, diluted to 500 parts with water and allow that to well penetrate the wood. Let this coat dry about 6 hours, or over night; then apply a heavy coat of hot, raw linseed oil. Allow to stand 6 hours, or over night, and scrub well with soap and water until all surplus colour has been removed, that is until the water stays clean; now allow to dry and rub down well with linseed oil, applying several coats (a day or two apart) if necessary to completely fill the pores of the wood. This gives a deep black finish, with a slight gloss, which can be kept in perfect condition by an occasional scrubbing with soap and water, and a subsequent rub-down with linseed oil.

**Respirators, Military, Solution for Saturating.** (*Chem. & Drugg.*, 1915, 86, 727.) The hoods, with the insertion for the eyes, are saturated with the following solution by means of a powerful spray; they are then packed in waterproof tissue to retain their moisture as long as possible, though the War Office says that it is not essential if water is at hand; it must be moist to be efficient, the nose and mouth being covered. The eye piece is first stitched in, then adhesive plaster placed over it. Sodium hyposulphite, 15 oz.; sodium carbonate, 5 oz.; glycerin (by weight), 2 oz.; water, 10 oz. Mix the glycerin and water, and dissolve the salts in the mixture, straining if necessary. A little eucalyptus oil may be added to the solution as a refresher. About 6 fl. dr. of the solution is used for each respirator.

**Secret Preparations, The Debt which Medicine Owes to.** O. Raubenheimer. (*J. Amer. Pharm. Assoc.*, 1915, 4, 373.) Many preparations are in daily use as household remedies, and even as official preparations, which originated as secret medicines. A number of the older remedies are still extensively used to-day, in spite of the fact that some of them were patented almost two hundred and fifty years ago. Physicians and pharmacists have lent a helping hand in originating preparations which have greatly enriched our *Materia Medica*, for instance: Paregoric was originated by Dr. Le Mort, Professor of Chemistry at the University of Leyden. Wine of opium originated by the celebrated English physician, Sydenham. Comp. powder of rhubarb originated by Dr. James Gregory, Professor of Medicine at Edinburgh. Calamine ointment originated

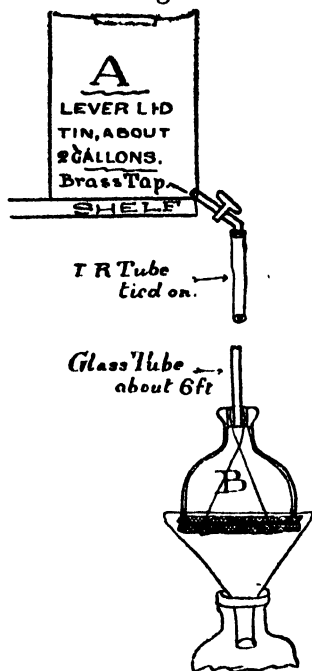
by Dr. Daniel Turner, a celebrated surgeon at London. Powder of ipecac. and opium, by Thomas Dover, doctor and pirate. Solution of potassium arsenite, by the apothecary, Thomas Fowler. Fowler originally kept its arsenic content secret and named it Mineral Solution. Perhaps the most important of this class is Rochelle salt, which was accidentally discovered by the French apothecary, Pierre Seignette, in 1672, and the composition of which was kept secret for almost sixty years, until at last, in 1731, two French pharmacists made an analysis. Sodium bicarbonate was introduced into medicine by the Berlin apothecary, A. W. Bullrich, as his "Universal Salz" in 1840.

**Shampoo Jelly.** (*Nat. Drugg.*, 1915, 45, 154.) Castile soap, in shavings, 8 oz.; potassium carbonate,  $1\frac{1}{2}$  oz.; water, 12 oz.; honey, 2 oz.; glycerin, 2 fl. oz.; oil of lavender flowers, 10 drops; oil of bergamot, 22 drops. Heat the three first ingredients, preferably on a water-bath, until a homogeneous mixture results; then add the honey and glycerin, and when cold, incorporate the oils. Should the mixture be too firm, warm it slightly and add a sufficient quantity of lukewarm water to reduce it to the proper consistency. Preserve in tightly-sealed containers.

**Silver Polish in Cake Form.** (*Nat. Drugg.*, 1915, 45, 23.) Magnesium carbonate, 1 oz.; prepared chalk, 1 oz.; fuller's earth,  $\frac{1}{2}$  oz.; dextrin, 30 grains; water, enough to make a paste. Put in tin boxes and allow to dry. To use, moisten a cloth with dilute  $\text{AmOH}$  and rub on the cake of polish; then apply to the article to be polished.

**Simple Pressure Filter for Thick Liquids.** M. Firth. (*Pharm. J.*, 1914 [4], 39, 485.) The following is a simple and cheap method of filtering all refractory liquids, including glycerin 100 per cent., syrups, oxymels. The filter, *B*, is made by cutting off the top of a quart bottle with a wheel glass-cutter. This is easily accomplished by standing the bottle against a block of wood of the correct height, and whilst holding the cutter on the top of the block, turning the bottle against it, so as to make a scratch quite round. Then turn the bottle round and round against the sealing jet, and the top will crack off with a clean edge. Now tie a piece of gamgee tissue very tightly over the open end of the cut-off top, fit a glass tube about 6 ft.

long, with a very tight cork, and the filter is ready. It is safer to tie a string once or twice around the neck and under the



"gamgee," as indicated in the sketch, to prevent the filtering pad slipping off with the pressure. To make the filter closer, tear up a grey filter paper, pulp it by shaking with hot water in a bottle, and pour into the filter whilst it is standing on a flat surface; allow the water to drain away, and remove the rest of the water by blowing through the bottle neck. The tin container is drawn out of scale for convenience. The tap is an ordinary gas fitting, which any plumber will fit. Two gallons is a convenient size. The rubber tubing need not be more than a few inches long, and the joints to tap and glass tube must be securely tied. This being a pressure filter, the speed, of course, depends on the length of the tube; 6 ft. is usually sufficient,

the container being on a shelf and the receiver on the floor.

**"Skin Foods" and Toilet Creams.** H. C. Bradford. (*Drugg. Circ.*, 1915, 59, 221.) The so-called "skin foods" are creams containing a considerable proportion of animal fat. *Skin Food*.—Oil of theobroma, 8 oz.; hydrous wool fat, 8 oz.; cotton seed oil, 2 oz.; boric acid, 4 drachms; simple tincture of benzoin, 4 drachms. Melt the solid fats on the water-bath; rub the boric acid smooth with a portion of the melted mixture; add to the melted fats and add the oil slowly with constant stirring. Then add the tincture in the same manner; remove from heat, and beat briskly until cold and the product is light and fluffy. Transfer to jars and allow to stand open for 24 hours. Gloss the top by holding momentarily near a source of heat. *Witch Hazel Skin Food*.—Hydrous wool fat, 24 oz.; oil, 6 oz.; distilled witch hazel extract, 2 oz. Mix in the usual manner. *Lotus Skin Food*.—Spermaceti, 10 oz.; white bees-

wax, 8 oz.; hydrous wool fat, 8 oz.; coconut oil, 8 oz.; oil of theobroma, 4 oz.; cotton seed oil, 24 oz.; water, 14 oz.; borax, 2 drachms; simple tincture of benzoin, 4 drachms. Mix in the usual manner, using an ice-cream mixer and beating vigorously. *Madame de Compierre's Beauty Cream*.—Spermaceti, 1; mutton tallow, 8; hydrous wool fat, 9; coconut, oil, 8; expressed oil of almonds, 8. Melt on a water-bath, mix well and pour into jars. *Ivory Cerate*.—White wax, 4; spermaceti, 2; oil of theobroma, 2; culinary cotton seed oil, 4 to 8. Mix, melt and mould. The amount of the oil is varied to suit the temperature and conditions. "*Cocoa Butter*" *Brick Massage Cream*.—Oil of theobroma, 16; white wax, 1; coconut oil, 1. Mix, melt and mould. *Purification of Cacao Butter and other Solid Fats*.—Prepare some good strong lime water. Slice the rancid fat into wafers, or, what is better, by means of a grater or chopper, reduce it to a coarse granular powder. The smaller the pieces, the quicker and better the lime will act. Immerse these chips in the lime water, and let them remain about 24 hours, stirring occasionally, and taking care that they are at all times submerged. Then drain off the water, and if any trace of the volatile acids remain, repeat the process. This done, wash thoroughly in clear water to remove all traces of lime, drain well, melt on a water-bath, and continue the heat until the water adhering to the fat has been driven off. Then cast into cakes in the usual manner. *Camphor Ice*.—Paraffin, 2; white wax, 2; white petrolatum, 12; camphor, 3. Melt the first three ingredients on a water-bath; add the camphor in powder, and continue to heat, with stirring, until it is dissolved. Then pour into moulds. *Perspiration Cream*.—White wax, 8 oz.; liquid petrolatum, 24 oz.; borax, 100 grains; benzoic acid, 20 grains; salicylic acid, 400 grains; hot water, 6 oz. Melt the wax and oil and heat to about 160°F. Dissolve the other materials in the water, heat to the same temperature as the wax solution, and pour it into the latter, beating briskly until the cream is formed. Here a comparatively high temperature of the solutions, plus a small amount of stirring, results in the peculiar, beautiful enamelled or glossy cream. "*La Rouche*" *Bath Cream*.—Tannic acid, 4; expressed oil of almonds, 160; hydrous wool fat, 240. Melt, mix and beat until smooth. "*Queen Draga's*" *Complexion and Pimple Cream*.—Artificial musk, 1; coumarin, 5; ichthyol, 150; white petrolatum, 2,500. Mix well, adding the ichthyol last. "*Pacific*"



**Wrinkle Cream.**—Simple tincture of benzoin, 1 drachm ; spirit of camphor, 1 drachm ; orange flower water, 1 drachm ; gelatin, 4 drachms ; powdered alum, 15 grains ; glycerin, 2 oz. ; mutton suet, 8 oz. Dissolve the alum in the orange flower water, add the gelatin, and soak until it is thoroughly softened. Add the glycerin and heat on the water-bath until the gelatin is dissolved. Melt the suet, add it to the solution very slowly and with constant stirring. Follow with the remaining ingredients in the same manner. Then remove from heat and with an egg beater whisk until the cream is cold and fluffy. It is directed to be applied to the wrinkles at night and well massaged into the skin for 10 to 15 minutes.

**Smelling Salts, Filling for Bottles of.** S. A r t h u r. (*Pharm. J.*, 1914 [4], 39, 691.) The use of washing soda, in small crystals, moistened with strong AmOH solution, and suitably perfumed and coloured, is suggested as a satisfactory substitute for the usual ammonium carbonate.

**Spas and Baths, English.** J. A. T h o m a s. (*Pharm. J.*, 1914 [4], 39, 660.) A short description of the thirteen chief English mineral water spas and baths, with the addresses of officials at each, from whom details may be obtained.

**Stearin Creams.** H. C. B r a d f o r d. (*Drugg. Circ.*, 1915, 59, 360.) These preparations are known as vanishing, disappearing or greaseless creams and are popular toilet articles. **Stearin-Borax Emulsion.**—Borax, 5 oz. ; stearin (granulated), 8 oz. ; distilled water, 3 pints 4 fl. oz. ; perfume and colour, of each a sufficient quantity. Dissolve the borax in the water, and heat the solution on a water-bath to about 100°C. Add the stearin and stir vigorously until it is melted, and thoroughly incorporated with the solution. Then remove from the water-bath, and continue the stirring until the product is thoroughly "set" and cold. The perfume and colour should be added when the product has cooled down to about 60° or 70°C., and thoroughly beaten in. As soon as the product is cold, transfer to containers which should be as nearly airtight as possible. If ordinary jars or boxes are to be used, run a thin layer of melted paraffin on top of the cream. This will preserve it perfectly until the jar is opened. The above formula represents the simplest of the emulsion processes, and the product is highly satisfactory, as well as cheap. So far as usefulness

goes, it is just about as good as any stearin cream. The great drawback to this is its lack of keeping qualities. Packaged in tubes, it does very well, and jars handled as above, will keep it for a reasonable time, but it is not permanent. The next formula is better in this respect, although the product is not entirely permanent.

*Stearin-Petrolatum Emulsion.*—Stearin, 9 oz.; liquid petrolatum, 4 oz.; powdered borax, 2 drachms; potassium hydroxide, 137 grains; distilled water, 43 oz.; perfume and colour, of each a sufficient quantity. Melt the stearin on a water-bath, add the liquid petrolatum, and stir until thoroughly combined. Dissolve the KOH in half the water, heat the solution to about 100°C., and pour it slowly, in a thin stream, into the latter, stirring vigorously all the while. Dissolve the borax in the remainder of the water, heat the solution and add to the other mixture exactly as the potassium hydroxide solution was added. Remove from heat and continue the stirring, or beating, until the product is cold. The perfume and colour should be added in the same manner as previously directed. This is not a true "greaseless" cream, but it does all the work of these, and the additional oil makes hardly a discoverable difference in the after-effects of the application.

*Stearin Soap Cream—No. 1.*—Stearin, 16 oz.; powdered borax, 8 oz.; glycerin, 8 oz.; monohydrated sodium carbonate, 765 grains; distilled water, 96 fl. oz.; colour and perfume, of each a sufficient quantity. Put the water, glycerin, borax and sodium carbonate into a kettle on a water-bath and raise to the boiling point, stirring until solution is complete. The stearin should be granulated, then add it slowly and with constant stirring, to the hot solution. Continue the heat and stirring, until the whole thing becomes an oily-looking, smooth, semi-transparent liquid. Remove from the heat, and keep the beater or stirrer going until the product is cold, adding the colour and perfumes, in the manner directed under previously given formulæ, or when the product has cooled to 60° or 70°C. *Stearin Soap Cream—No. 2.*—Stearin, 8 oz.; glycerin, 8 oz.; boric acid, 8 grains; potassium carbonate, 360 grains; distilled water, 40 oz.; colour and perfume, of each a sufficient quantity. The manipulation of the above ingredients is as in the previous formula. Mix all the materials except the stearin, the perfume and the colour; heat to the boiling point on the water-bath. add the stearin, previously granulated, or at least cut and

broken into small pieces, continue the heat and stirring until saponification is complete (indicated by the oily appearance and semi-transparent look of the product), then remove from heat, and stir until cold, beating in the perfume and colour in the manner already directed. *Stearin Soap Cream—No. 3. "Peroxide."*—Stearin, 6 oz. ; anhydrous wool fat, 1 oz. ; glycerin, 6 oz. ; solution of hydrogen dioxide, 1 oz. ; water, 32 oz. ; monohydrated sodium carbonate, 5 drachms ; borax, 2 drachms ; perfume and colour, of each a sufficient quantity. Mix the water and glycerin, and heat to about 90°C. on a water-bath. Add the borax and sodium carbonate and stir until they are dissolved. Melt the stearin and wool fat together, and raise the temperature to about 90°C. Then pour the borax-soda solution, slowly, in a thin stream, into the mixture of melted fats, keeping the stirrer going vigorously all the while. Continue the heat and stirring until effervescence has ceased, and the saponification is complete, then let cool down to about 40° to 50°C. ; add the hydrogen dioxide solution, the perfume and the colouring, and continue to beat until cold. This is a good example of a "peroxide cream." The amount of the latter ingredient could be increased if desired, but it is best to use caution in doing so. Hydrogen dioxide solution does not seem to work as well in practice for such purposes as this, as a theoretical study of the matter would lead one to think. The acid—which it is apt to contain—is prone to redden and roughen the skin, so that its use is limited to that of a skin bleach. *Stearin Soap Cream—No. 4.*—Stearin, 30 Gm. ; cacao butter, 5 Gm. ; sodium carbonate, 20 Gm. ; powdered borax, 5 Gm. ; glycerin, 25 c.c. ; mucilage of acacia, 100 c.c. ; water, 400 c.c. ; colour and perfume, of each q.s. Mix the water, mucilage, borax, and  $\text{Na}_2\text{CO}_3$  and heat on the water-bath until dissolved. Melt the stearin and cacao butter together, and pour very slowly into the hot aqueous solution with constant stirring. Continue heating and stirring until effervescence ceases and saponification is complete ; then remove from the heat and stir until cold, adding the colour and perfume during cooling. *Stearin Soap Cream—No. 5. Witch Hazel Foam.*—Granulated stearin, 100 Gm. ; sodium carbonate, 5 Gm. ; glycerin, 15 Gm. ; distilled extract of witch hazel, 500 Gm. ; distilled water, to make 1,000 Gm. Mix the  $\text{Na}_2\text{CO}_3$  and glycerin with 500 Gm. of water, heat on water-bath until dissolved. Add the stearin, and continue stirring until saponification is complete. Remove

from the heat, and when the temperature has fallen to 80° or 70°C. add the witch hazel extract and continue vigorous stirring until cold. In preparing these creams, it is important that the stearin used should be of good colour and free from rancidity. For stirring a hard wooden paddle is to be preferred. Only distilled water should be used. Once started, the process should be continuous and stirring not intermitted. Care should be taken to use capacious vessels on account of the effervescence.

The peculiar "fluffy" texture of these creams is easily attained by beating air into the mass. The cream is allowed to stand overnight and beaten up in the morning. This may be done in an ice-cream whisk or mechanical beater, the jacket being first filled with warm water and the softened cream beaten up to twice its volume. Then cold water is run into the jacket and the beater kept going until the mass is perfectly cold.

**Sulphur Skin Lotions.** (*Nat. Drugg.*, 1915, 45, 23.) (1) Precipitated sulphur, 36 gr.; prepared calamine, 120 gr.; glycerin, 1 fl. oz.; solution of carmine, 20 minims; rose water, enough to make 8 fl. oz. (2) Carmine, 2 gr.; precipitated sulphur, 60 gr.; zinc oxide, 120 gr.; zinc sulphocarbolate, 20 gr.; eau de cologne, 6 fl. drachms; glycerin, 6 fl. drachms; rose water, enough to make 6 fl. oz.

**Summer Beverages.** (*Pharm. J.*, 1914 [4], 39, 46.) *Lemonade Powders.*—Castor sugar, 8 lb.; tartaric acid, 7 oz.; oil of lemon, 2 drachms 20 minims; soluble saccharin, 30 grains; tartrazine, 16 grains. Mix intimately and pack 1½ oz. quantities into envelopes. The contents of each packet make a pint and a half of excellent lemonade. The envelopes should be yellow in colour. *Gingerette Powders.*—Gingerin, 20 grains; tartaric acid, 2 oz.; oil of lemon, 30 minims; soluble saccharin, 25 grains; Bismarck brown, 6 grains; castor sugar, 4 lb. Divide into 1½ oz. powders and pack with similar directions to the lemonade powder. *Lemon Syrup.*—Citric acid, ½ oz.; tincture of lemon, 6 drachms; sugar, 1 lb.; boiling or hot water to 2 pints.

**Tar Soap, Liquid.** (*Pharm. Zeit.*; *Nat. Drugg.*, 1914, 44, 294.) Mix 200 Gm. of tar with 400 Gm. of oleic acid, warm the mixture slightly, and filter. Then warm the filtrate on a water-bath and neutralize the acid by adding an alcoholic solu-

tion of KOH. Next add 100 Gm. of EtOH, a small amount of olive oil, and bring the weight of the finished product up to 1,000 Gm. by the addition of glycerin.

**Tollet Creams.** H. C. Bradford. (*Drugg. Circ.*, 1915, 59, 153.) *Cold Cream*.—Liquid paraffin, 96 fl. oz.; white wax, 30 oz.; water, 32 fl. oz.; borax, 1 oz. Dissolve the borax in the water; melt the wax, add the oil, and bring to a temperature of about 200°F., or even a little more; heat the sodium borate solution to the same temperature and pour it into the hot oil solution with vigorous stirring. It is important that the solutions should be both heated almost to boiling, as directed above, and then mixed, a comparatively slight amount of vigorous stirring—not beating—to make a snow-white cream with a shining, enamelled appearance, and is light and fluffy. If the two solutions are heated only enough to ensure mixing, and then mixed in the manner directed, the product is only a dull, heavy, sodden mixture. The above recipe may be cheapened in cost, without in any way reducing the quality, by replacing about half the mineral oil with an equal volume of the cotton seed oil and by using about 6 oz. of paraffin instead of an equal weight of the-white wax.

*Theatrical Cold Cream*.—Oil, 6 pints 8 fl. oz.; hard paraffin, 1 lb.; white wax, 3 lb.; borax, 3 oz.; water, 6 pints 8 fl. oz. Mix as directed in the preceding recipe. These two formulæ are very similar, and the cost of the two is about the same. Any variety of oil may be used, or a mixture may be employed.

“*Make-up*” *Cold Cream*.—Fresh sweet lard, 10 oz.; castor oil, 4 oz.; spermaceti, 2 oz.; borax, 20 grains; water, 1½ oz. Melt the spermaceti, add the lard, and then the castor oil. Dissolve the borax in the water; heat both solutions to about 150°F. and pour the aqueous solution into the mixture of fats, beating briskly until the product is nearly cold. This product is very effective, removing grease paint and theatrical make-up from the skin.

*Oxygenated Cold Cream*.—Paraffin, 250 Gm.; white wax, 250 Gm.; oil, 1,000 Gm.; sodium perborate, 10 Gm.; water, 380 Gm. Mix in the usual manner, except that here the heat should be no more than enough to keep the mixture of wax and oil fully liquefied, while the aqueous solution of the perborate should be warmed to the same temperature. Pour the latter into the former, slowly, beating briskly, and continue until

the product is cold. This continued beating makes the cream light and fluffy.

*"Cocoa Butter" Cold Cream.*—White wax, 6 oz.; paraffin, 4 oz.; spermaceti, 10 oz.; oil, 80 oz.; oil of theobroma, 16 oz.; sodium borate, 4 oz.; water, 60 oz. Mix in the usual manner, and beat with an egg beater, or better, in an ice-cream freezer to make light and fluffy. The cream made by this recipe is excellent, and reasonable in cost. If vegetable oil be employed, it will serve as well for "skin food."

*"Satin" Cream.*—Pure sweet unsalted lard, 220 Gm.; potassium hydroxide, 31 Gm.; alcohol 60 per cent., 10 Gm.; water, 90 Gm. Dissolve the potassium hydroxide in the water and if there is any sediment, let the fluid stand until it settles, and pour off the clear solution. Put this along with the lard into a warm pan and mix together thoroughly, working in the alcohol in portions. With the above proportions a product stiff enough for a jar is obtained; but by increasing the alcohol it can be made into a rather thick liquid, with a beautiful effect, when shaken, like liquid pearl. Applied freely, and wiped off with a piece of cloth or gauze, it will remove soot, dirt, dust, travel stains, and all else similar, from the face and hands, much easier, quicker, and more gently than the most vigorous scrubbing with soap, and added to this, the trace of fat left behind will be an added protection to the skin.

*Cucumber Cream.*—White wax, 9 oz.; oil, 24 oz.; benzoic acid, 15 grains; cucumber juice, 10 oz. Mix in the usual manner, the juice taking the place of the water in the ordinary formula. The benzoic acid is superfluous, and can be omitted if the juice be made properly, as directed below.

*Cucumber Juice.*—Take nice large green cucumbers; do not wash, but wipe off all dirt and dust with a damp cloth. Then put through an ordinary food or meat chopper, using a rather fine knife. Collect the pulp in an enamelled pan and heat almost to boiling, then transfer to a straining bag, and let drain until dry. Express the bag thoroughly, but do not mix the expressed juice with that which came through by dropping. To each gallon of this latter add 1 pint of alcohol, in which has been dissolved 2 oz. of benzoic acid. Mix thoroughly, let stand 24 hours and filter. Collect the filtrate and preserve it in well corked bottles. The juice expressed from the pulp is subjected to the same treatment, except that it should first be heated to boiling and allowed to boil about 1 minute, then passed

through a muslin strainer to precipitate the mucilaginous matter.

**Tollet Powders and Lotions.** E. W. Lucas. (*Perfum. Record*, 1914, 5, 280.)

**VIOLET POWDERS : NON CLINGING TYPES.**—(1) Starch powder, 890 ; orris root powder, 100 ; oil of neroli, 5 ; oil of bergamot, 3 ; otto of rose, 2. (2) Starch powder, 500 ; kaolin, 480 ; synthetic musk, 5 ; oil of bergamot, 12 ; oil of clove, 3. This is a cheaper form.

**CLINGING TYPES.**—(1) Kaolin, talc, zinc oxide, wheat starch, of each, equal parts. (2) Prepared white diatomite, 50 ; zinc oxide, 25 ; talc, 25. (3) Talc, 2 ; kaolin, 1 ; bismuth oxychloride, 1. (4) Zinc oxide, magnesium carbonate (light), kaolin, wheat starch, of each, equal parts. (5) Bismuth oxychloride, 1 ; zinc oxide, 6 ; prepared white diatomite, 5 ; talc, 8. (6) Zinc stearate, prepared white diatomite, bismuth oxychloride, talc, of each, 5. (7) Soft white paraffin, 1 ; elutriated diatomite, 10 ; talc, 9. Dissolve the paraffin in a little hot  $\text{CHCl}_3$  or petroleum ether, and spray it upon the mixed powders, stirring rapidly meanwhile. When the whole of the paraffin has been added, spread the powder in a thin layer for the solvent to evaporate. Some recommend lanolin in place of the paraffin, but the odour is unpleasant, and is quite difficult to cover.

*Prepared White Diatomite.*—For this good white kieselguhr or diatomite is dried, thoroughly ground, and sifted through bolting cloth. If the material is ground in a disintegrator, the lighter particles that collect in the "balloon" make an excellent basis.

*Colours for Face Powders.*—These must be added in the wet state. When carmine is used it must be of the best quality, and it should be ground in with a little dilute ammonia solution. In any case sufficient water must be used to make the mixture quite wet. For flesh tints plenty of yellow must be used. Some makers use cadmium sulphide, but it is rather too bright, and yellow ochre is generally preferable. *For Flesh Tint*—(1) Yellow ochre, 90 ; bole, 6 ; carmine, 4. (2) Yellow ochre, 90 ; bole, 3 ; hydrated ferric oxide, 2 ; carmine, 5. *For Pink Tint*—Yellow ochre, 75 ; carmine, 25. *For Cream or Rachel*—Yellow ochre, 94 ; bole, 4 ; burnt sienna, 2. Of the foregoing concentrated tinting powders from 60 to 120 grains are required for each pound of white face powder.

*Perfuming the Powder.*—If the concentrated floral extracts are employed, from 10 to 15 drops per lb. are sufficient. The perfume must be well stirred in, and the powder kept for a little time to allow it to become thoroughly permeated. Should it not be desirable to employ floral perfumes, the following volatile oils may be blended, care being taken that none predominates—otto of rose, bergamot, geranium, ylang ylang, neroli, patchouli (the merest trace). Of these not more than 12 drops in all per lb. will be required. Synthetic perfumes are sometimes used where cost is an important consideration; for example, artificial musk or musk ambrette, ionone, vanillin, coumarin, aubepine, heliotropin, etc. These are very permanent, and only a grain or two of each per lb. of basis will be required.

*Cake Powders.*—These consist of toilet powders made damp with tragacanth mucilage 2 per cent. and afterwards pressed into moulds. The cakes must be allowed to dry very gradually. Some makers add a trace of plaster of Paris, about 2 per cent., before moistening. This makes a firmer cake, but it is not so pleasant to use.

*Nursery Powders.*—The best types contain boric acid, zinc oxide and starch. The *ideal* nursery powder should not contain any natural earthy matter as fuller's earth, talc or kaolin, unless previously sterilized. A tetanizing bacillus is frequently present in the soil, and not a few cases of tetanus have been traced to the use of unpurified fuller's earth on excoriated surfaces. If talc, kaolin and fuller's earth are used, the powders should be boiled for 20 minutes with water, allowed to deposit, the deposit collected and dried. No admixture of germicides that could be borne on the skin has the slightest effect on bacilli or their spores contained in natural earths.

In the following formulas each article must be in very fine powder and quite dry, and the finished mixture sifted by shaking through bolting cloth. (1) Zinc oxide, boric acid, starch, of each, equal parts. (2) Boric acid, 1; zinc oxide, 1; sterilized talc, 2. (3) Pure carbolic acid, 5; soft white paraffin, 50; boric acid, 290; zinc oxide, 200; starch, 455. Dissolve the carbolic acid and paraffin in a little hot petroleum ether and distribute it on the starch. Mix in the other powders, and expose to the air for the solvent to evaporate. Perfuming nursery powders should be carried out with discretion. The odour produced should be faint and delicate. One or two drops of rose oil or of concentrated floral extract per lb. is quite sufficient.



*Antiseptic Foot Powder.*—Boric acid, 75 ; zinc oxide, 5 ; sterilized talc, 20. Oil of eucalyptus or thyme oil may be added as perfumes.

*SKIN LOTIONS.*—*Milk of Cucumber ; Milk of Roses.*—(1) Blanched sweet almonds, 1 oz. ; glycerin, 1 oz. ; simple tincture of benzoin,  $\frac{1}{2}$  fl. oz. ; oil of ylang ylang, 5 drops ; powdered borax, 1 drachm ; rose water to make 20 fl. oz. Add the borax to the blanched almonds and beat to a smooth paste. Gradually dilute with the rose water, straining through fine muslin until 15 fl. oz. is obtained. Add the tincture to the emulsion very gradually, shaking well after each addition ; lastly add the ylang ylang, with enough rose water to produce 20 fl. oz. If desired,  $\frac{1}{4}$  oz. of fresh curd soap, cut in fine shavings, may be dissolved in a portion of the rose water by the aid of gentle heat. This prevents separation. For "Milk of Cucumber" add 2 drachms of cucumber essence. (2) Powdered curd soap,  $\frac{1}{2}$  oz. ; powdered borax,  $\frac{1}{4}$  oz. ; cucumber pomade, 1 oz. ; oil of geranium, 20 drops ; glycerin, 1 fl. oz. ; water to produce 20 fl. oz. Mix the soap, borax and pomade ; gradually add enough water to form a smooth paste. Set aside for a few hours ; then gradually add the rest of the water, glycerin and perfume.

*Transparent Cucumber Lotion.*—Fresh cucumber juice, 20 fl. oz. ; rectified spirit, 5 fl. oz. ; orris powder, 1 oz. ; glycerin, 2 fl. oz. ; fresh spinach,  $\frac{1}{4}$  oz. ; otto of rose, 5 drops ; oil of neroli, 5 drops. Set aside for a week, with occasional shaking, and filter. [If spinach is not in season a suitable tint may be obtained by means of chlorophyll.—Ed. Y.B.]

*Lait Virginal.*—Simple tincture of benzoin, 6 drachms ; saponin, 10 grains ; glycerin, 2 fl. oz. ; rose water, orange flower water, elder flower water, of each, 6 fl. oz. Mix the waters, glycerin and saponin. To the solution, add the tincture of benzoin, poured in very slowly. This gives a milk-like lotion which is generally approved.

*Sunburn Lotions.*—A lotion to prevent sunburn, and also to counteract its effect, must contain glycerin. Ammonium chloride is also useful. The lotion to be described should be applied to the exposed parts before going out into the sun. If sunburn has already taken place, the lotion should be dabbed on and allowed to dry. (1) Ammonium chloride,  $\frac{1}{2}$  oz. ; lead acetate, 5 grains ; glycerin, 1 fl. oz. ; rectified spirit,  $\frac{1}{2}$  pint ; oil of neroli, 5 drops ; rose water, to produce 1 pint. Dissolve the

ammonium chloride and glycerin in half the rose water, and the lead acetate in the other half. Mix the solutions very slowly and add the rest of the ingredients. The trace of lead chloride formed dissolves in the excess of water; it helps to allay the smarting. (2) Zinc oxide,  $\frac{1}{2}$  oz.; borax,  $\frac{1}{4}$  oz.; glycerin, 1 fl. oz.; lavender water, 1 fl. oz.; distilled water, to produce 1 pint. This must have a "shake the bottle" label. The lotion must be dabbed on the inflamed surface and allowed to dry on; it also must be washed off with plain water before using soap.

*Freckle Lotions.*—Freckles are more easily prevented than removed. Remedies must be both alkaline and strongly alcoholic. The following lotion is one of the best: Sodium carbonate crystals, 1 drachm; glycerin,  $\frac{1}{2}$  fl. oz.; rose water, 5 fl. oz.; lavender water, to produce 1 pint. Mix, set aside for a week, and filter. The lotion must be gently rubbed over the freckles twice a day and allowed to dry on.

*Perspiration Lotions.*—These lotions are used for dabbing the armpits, between the toes, etc., to check excessive perspiration. They depend for their activity on the astringency of the alum. Take of glycerin of alum, 4 fl. oz.; rose water, to produce 1 pint; solution of cochineal, sufficient to colour. Directions.—Thirty drops to be dissolved in a wineglassful of water, and used for dabbing the armpits, between the toes, etc. After the application has been absorbed the parts must be well dusted with an antiseptic powder (e.g. boric acid 1 part, purified talc 2 parts).

*Buttermilk Lotion.*—Take  $\frac{1}{2}$  oz. of good quince seed and wash rapidly with cold water. Place the washed seed in 15 fl. oz. of distilled water, and simmer gently for 5 minutes. Strain through felt several times; add in the order named: Glycerin, 1 fl. oz.; tincture of quillaia,  $\frac{1}{2}$  fl. oz.; simple tincture of benzoin,  $\frac{1}{2}$  fl. oz.; oil of neroli, 5 drops; oil of ylang ylang, 3 drops; lactic acid, 1 fl. drachm; water, to produce 20 fl. oz.

*Liquid Enamel.*—Liquid enamels occupy a place midway between toilet powders and lotions. (1) Bismuth oxychloride,  $\frac{3}{4}$  oz.; glycerin, 2 fl. drachms; concentrated extract of violet (500), 5 drops; rose water, to produce 2 fl. oz. (2) Zinc oxide,  $\frac{1}{2}$  oz.; whitest talc,  $\frac{1}{4}$  oz.; eau de Cologne oils, 7 drops; glycerin, 2 fl. drachms; distilled water, to produce 2 fl. oz. If tinted enamels are required a trace of the tinting ingredients described under toilet powders may be added.

*Face-Bleach Lotion.*—Sodium sulphite,  $\frac{1}{4}$  oz.; oil of neroli

5 drops ; borax,  $\frac{1}{4}$  oz. ; glycerin, 2 fl. oz. ; elder flower water, to produce 20 fl. oz. The lotion to be dabbed on the skin before retiring.

**Skin Tightener.**—Zinc sulphate, 40 grs. ; rectified spirit, 2 fl. oz. ; quince seed,  $\frac{1}{4}$  oz. ; glycerin,  $\frac{1}{2}$  fl. oz. ; oil of ylang ylang, 5 drops ; rose water, 5 fl. oz. ; elder flower water, 5 fl. oz. ; distilled water, to produce 20 fl. oz. Wash the quince seed in cold water, drain and simmer the clean seed in 10 fl. oz. of distilled water for 5 minutes. Strain through felt. Add the other ingredients to the strained liquid. Set aside for 14 days, again strain and bottle off. A small quantity to be rubbed into the flaccid skin every night. After a few applications the skin should be well massaged with cold cream.

**Vinegar Eels, to Destroy.** J. P. Sacher. (*Chem. Zeit.*, 1914, 38, 1021.) Vinegar eels are killed by a few minutes' exposure to 45°C. in ordinary vinegar. They may also be killed by a few hours' exposure to direct sunlight, but this method cannot be used if further action of *Mycoderma aceti* is desired, as this ferment is also destroyed by sunlight. Vinegar eels are also destroyed if air is excluded from the container for 6 to 8 weeks, by sealing the vessel with hard paraffin. CO<sub>2</sub> has no effect on them. One per cent. of NaCl kills them, but 0.1 per cent. is ineffective. One per cent. of NaNO<sub>3</sub> kills them in 3 days ; but KNO<sub>3</sub> has no effect. Other Na salts are toxic to the eels, but not K salts. EtOH is only partially effective in killing the eels, even when used in large quantity.

**Wart Remover.** (*Nat. Drugg.*, 1915, 45, 23.) Salicylic acid, 5 ; boric acid, 15 ; calomel, 30. Mix, and make into a fine powder. Put into small glass tubes, with the direction to rub a small portion on the wart three times daily.

**X-Ray Work for Pharmacists.** F. Goldby. (*Pharm. J.*, 1915 [4], 40, 64.) Practical instructions on the installation and working of an X-ray apparatus are given. The following is the developer recommended : (No. 1) Metol, 10 grains ; hydrokinone, 120 grains ; citric acid, 10 grains ; sodium sulphite, 4 drachms ; potassium bromide, 30 grains ; distilled water, to 10 fl. oz. (No. 2) Potassium hydroxide, 160 grains ; distilled water, to 10 fl. oz. The above is used in equal parts of 1 and 2, and is of double strength. Development is effected in the ordinary ruby light of the dark-room, and should be fully carried

out, care being taken not to under-develop. The resulting negatives must be examined from the film side, using a strong but well diffused light. If prints are required they may be made upon ordinary P.O.P., or glossy gaslight paper, and it will be necessary to remember that as the negative is a "shadow-graph," the prints will be laterally reversed. It is only occasionally, however, in cases of private patients, that prints are required—as a rule the surgeon is quite satisfied with the negative for examination. The article is illustrated with reproductions of some fine skiagrams.

**Yerba Santa Cough Mixture or Compound Syrup of Yerba Santa.** (*Nat. Drugg.*, 1914, **44**, 503.) Yerba santa, 2 oz. ; grindelia, 1 oz. ; wild cherry, 1 oz. ; licorice root, 1 oz. ; ammonium bromide, 1 oz. ; pine tar,  $\frac{1}{2}$  oz. ; sugar, 16 oz. ; glycerin, 4 fl. oz. ; alcohol 90 per cent. and water, sufficient of each. Mix the four drugs, reduce them to coarse powder and extract by percolation, using first a menstruum of the glycerin and 8 fl. oz. each of alcohol and water, and then followed by diluted alcohol 49 per cent. until 22 fl. oz. of percolate are obtained. To this add the ammonium bromide and tar, macerate a few hours, agitating occasionally, filter, and in the filtrate dissolve the sugar by agitation and strain if necessary.

## RESEARCH LIST, 1915

THE following subjects are suggested for investigation. The Executive Committee hope that Members of the Conference will undertake to work on one or more of these. It should be noted that some of the subjects may have been appropriated already. In order to avoid duplication the Honorary General Secretaries trust that members will communicate to them their intention of working at any of the subjects mentioned; they also wish to direct attention to the fact that a special fund exists to defray expenses connected with research work. The Executive Committee will be glad to receive applications from members for grants from this fund.

*Apiol*.—A standard formula for its preparation is required.

*Atropine Sulphate*.—Is the commercial article variable in character?

*Belladonna Root*.—In what respects, if any, does a tincture from the fresh root differ in its composition and action from one from the dried root?

*Bismuth Phenate*.—An examination of commercial samples would be of interest. (Already undertaken.)

*Calx Sulphurata*.—An examination of the processes of manufacture and the purity of commercial samples is needed. (Already undertaken.)

*Cannabis Indica*.—The physical characters and therapeutic value of the official preparations are stated to be liable to considerable variation. An investigation is required to determine whether any chemical standard is possible, and if not, whether physiological tests should be introduced. A report on the comparative values of the official Indian drug and those varieties produced in Goa, Africa, America, and Greece is desirable.

*Casein Foods*.—A comparative examination of the so-called "Foods" or "Nerve Tonics" of the type represented by the combination of soluble casein and glycerophosphates, etc., would be useful.

*Casein (Soluble).*—A process is required for the preparation of a soluble casein.

*Drugs.*—The following drugs require further systematic investigation: *Cereus grandiflorus*, *Cassia fistula*, *Serenoa serrulata*, *Arnica montana*, *Monsonia ovata*, *Monsonia biflora*, *Thuja occidentalis*, *Tanacetum vulgare*, *Senecio jacobea*, *Achillea millefolium*, *Aletris farinosa*, *Cascara Sagrada*, *Senega*, *Senna* fruit.

*Ergot.*—A re-investigation of the pharmacy of this drug in the light of recent chemical work is required, and a method of determining the activity of the galenical preparations.

*Ferments.*—The action of ferments in inducing changes in galenical preparations might be studied.

*Formulæ.*—Improved formulæ are required for the administration of nauseous drugs, such as cascara, coca, etc.

*Galenicals.*—Investigation is required of the changes in the strength of galenicals, etc., during preparation and on keeping, as may render the original formula an unfair criterion of the finished product, e.g., loss of ammonia in filtering Tinct. Quininae Ammoniata; loss of formaldehyde from the tablets; loss of iodine in making Syrup. Ferri Iodidi.

*Gum-Resins.*—The value of the saponification numbers in determining the identity and purity of the resin of gum-resins.

*Liquor Hamamelidis.*—What is the nature of the aldehydic constituent in this preparation? (See *Year-Book*, 1911, page 195.)

*Male Fern.*—The chemistry and pharmacy of this drug both require investigation. (Already undertaken.)

*Mercury Zinc Cyanide.*—Is any change produced when dressings containing this substance are sterilized by heat?

*Morphine.*—Can the process described in the *Year-Book of Pharmacy*, 1907, page 107, for the determination of morphine be applied to opium and its preparations?

*Oil of Soya Bean.*—Can this be utilized in pharmacy?

*Opium, Extract of.*—To what is the loss of morphine due in making this extract? Is it constant with different lots of opium?

*Paraffin, Liquid.*—Suggestions are invited as to the best means of administering this article internally.

*Pareira (Bahia).*—Examination of the alkaloidal constituents is required.

*Phenol, Liquefied.*—The pharmacy of this substance requires further investigation.

*Pills.*—A systematic examination is required to determine the

time necessary for the solution or disintegration of pills prepared with different excipients and kept for various periods.

*Powdered Drugs*.—A systematic microscopical examination of powdered drugs is required.

*Quillaia Bark*.—Experiments are desirable to determine the best solvent for exhausting this bark for the purpose of making emulsifying agents, and a comparison of the official bark with the thin bark at present in commerce.

*Santonin*.—Analyses are required showing the percentage of santonin in Colonial and Indian species of *Artemisia* allied to *Artemisia maritima*.

*Saponins*.—A simple and accurate method of determining saponins in drugs is required.

*Solvents*.—Experiments are needed with a view to extending the use of solvents such as acetone, carbon tetrachloride, dichloroethylene, petroleum ether, amyl acetate, etc., in pharmacy.

*Strophanthus*.—An examination of the published methods of separating the different active principles obtained from the official seeds is needed with a view to recommending a standard process. The seeds in commerce are frequently mixed. Further information is required as to the active principles they severally contain.

*Tannin*.—A ready and tolerably accurate method for the determination of the tannin in various astringent drugs is required. (Already undertaken.)

*Taraxacum Root*.—The investigation of fresh drugs such as this by Bourquelot's method for the detection and isolation of easily hydrolysed glucosides is required.

*Tinctures (Concentrated and Non-Alcoholic)*.—The best methods of preparation and the examination of commercial samples are required.

*Valerian Root*.—Chemical investigation of the fresh root by Bourquelot's method is required.

THE TRANSACTIONS  
OF THE  
**British Pharmaceutical Conference**  
AT ITS  
***FIFTY-SECOND ANNUAL MEETING***  
HELD IN LONDON,  
**JULY 14, 1915.**



# British Pharmaceutical Conference.

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**FIFTY-SECOND ANNUAL MEETING, IN LONDON, July 14, 1915.**  
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# Programme of Conference Sessions

**The Lecture Theatre, 17, Bloomsbury Square,  
London,**

**WEDNESDAY, JULY 14, 1915.**

## *Order of Business.*

Address of Welcome.

Presidential Address by Major E. SAVILLE PECK.

The Honorary Treasurer's Report.

Report of the Executive Committee.

Election of Officers for 1915-16.

Invitation to Meet at Liverpool in 1916.

Vote of Thanks to the Pharmaceutical Society.

## THE CONFERENCE SESSION

### ORDER OF BUSINESS

*Wednesday, July 14, 1915*

The Conference Session opened at 11.0 a.m. in the Lecture Theatre of the Pharmaceutical Society of Great Britain, 17, Bloomsbury Square, London. Major E. Saville Peck (President) occupied the Chair, and was supported by Messrs. N. H. Martin and E. M. Holmes (Past Presidents of the Conference), Mr. P. F. Rowsell and Mr. R. R. Bennett (Hon. General Secretary).

### ADDRESS OF WELCOME

Mr. P. F. ROWSELL said that at a few moments' notice he found himself in an unexpected position. The President of the Pharmaceutical Society was detained by an important engagement, the Vice-President was unable to be present, and therefore, at the request of the Secretary of the Society, it fell to him to offer, on behalf of the Pharmaceutical Society of Great Britain, a very hearty welcome indeed to the British Pharmaceutical Conference. The Society is always anxious to welcome to its premises any section of pharmacists, any body of men, who are doing their utmost for pharmacy, and if any body of men had done something for the benefit of pharmacy, he ventured to say that it was the British Pharmaceutical Conference. He heartily welcomed to the premises of the Pharmaceutical Society of Great Britain this meeting of the British Pharmaceutical Conference.

The PRESIDENT said that on behalf of the British Pharmaceutical Conference, he wished to thank Mr. Rowsell most heartily for his very kind welcome.

### APOLOGIES FOR ABSENCE

Mr. R. R. BENNETT (Hon. Secretary) announced that apologies for absence had been received from Sir Edward Evans, Messrs. S. R. Atkins, W. Bates, F. W. Branson, H. Wippell Gadd, W.

Giles, E. F. Harrison, D. Hooper, Edmund Jones, W. A. H. Naylor, J. W. Richardson, T. Stephenson, J. O. Thomas, C. Symes, D. M. Watson, W. F. Wells, F. B. Wride, and the Registrar of the Pharmaceutical Society of Ireland.

LIST OF DELEGATES APPOINTED TO ATTEND THE MEETING.

*Pharmaceutical Society of Great Britain.*—The President, Vice-President, Messrs. F. E. Bilson, W. G. Cross, W. L. Currie, J. P. Gilmour, J. F. Harrington, P. F. Rowsell and the Secretary.

*Pharmaceutical Society of Great Britain (North British Branch).*—Messrs. W. B. Cowie, W. L. Currie, W. Giles, D. Gilmour, J. P. Gilmour.

*Brighton Association of Pharmacy.*—Mr. C. G. Yates.

*Bury and West Suffolk Chemists' Association.*—Messrs. O. A. Clark, A. G. Gamble.

*Devon Pharmaceutical Association.*—Mr. P. F. Rowsell.

*Dover Chemists' Association.*—Mr. J. H. Cuff.

*Edinburgh District Chemists' Trade Association.*—Messrs. J. T. Coats, J. Dick, A. Duncan.

*Exeter Association.*—Mr. P. F. Rowsell.

*Liverpool Chemists' Association.*—Messrs. A. C. Abraham, W. P. Evans, H. H. Jones, C. Symes, H. Wyatt.

*London, Eastern Association.*—Messrs. B. Abelson, A. R. Keith.

*London, North Eastern Association.*—Messrs. C. E. Fox, W. A. Scott.

*London, Northern Association.*—Messrs. A. W. Bromley, H. Skinner.

*London, South Eastern Association.*—Messrs. J. Milner, A. J. Wing.

*London, South Western Association.*—Messrs. E. A. Atkins, W. H. Goy.

*London, Western Association.*—Messrs. A. R. Melhuish, W. E. D. Shirtliff.

*Newport (Mon.) Pharmacists' Association.*—Mr. A. Gratte.

*Northumberland Association.*—Mr. G. Foggan.

*Scarborough and N. Riding Association.*—Messrs. E. R. Cross, G. Whitfield.

*Sheffield Pharmaceutical and Chemical Society.*—Messrs. S. Furnival, J. G. Jackson.

*Southampton Pharmaceutical Association.*—Messrs. W. Bates, H. Wilson.

*Sussex County Association.*—Mr. E. H. Farr.

## THE PRESIDENTIAL ADDRESS

### PHARMACY ORGANIZATION AND THE WAR

BY MAJOR E. SAVILLE PECK

When the Conference last year was bringing its session to a close, and its members were expressing their thanks to the pharmacists of Chester, there was already brooding upon the international horizon the dark cloud that was so soon to break and plunge practically the whole of Europe and many millions of men into the alembic of war. As to the origin of this conflict we know the simple surface facts—Austria declared war against Servia, Russia in consequence said she must stand by Servia, Germany declared she must support Austria, and France that she must back up Russia. Great Britain, upon Germany's refusal to give a satisfactory reply to her questions as to the maintenance of the neutrality of Belgium, was bound to enter the lists. Since the beginning of the war Turkey has joined forces with the Central Empires, and Italy has thrown in her lot with the Allies.

During the twelve months of bitter conflict every imaginable device that would either kill, maim, hinder, or unnerve the opposing foes seems to have been practised, the immediate issue is still undecided, and victory for the Allies is still long delayed. Men of the British Empire, both at home in the Mother Country and from her Dominions beyond the seas, have responded, voluntarily up to the present, to the call to arms, and we have thrown into the trenches troops worthy of Britain's best traditions. The Royal Navy, by its strength and vigilance, and with the assistance of the navies of our Allies, has kept open the highway of the seas and enabled a constant supply of food and raw materials to be maintained and distributed.

Notwithstanding this fact, such is the strength, the foresight, the efficiency, and the organization of the enemy that the whole resources of the Empire must be organized to supply men, material, and money, and that soon, if we would achieve an early victory. The National Registration Bill is certainly a step towards this national organization. For, as the President

of the Local Government Board stated on July 9, "What is wanted is something more than a census: information not only as to names and ages, but as to capacity, the training, and the suitability for various classes of work." It is hoped that the census will be followed up by efforts to enable both men and women to get that work where they can be of the greatest use to the nation as a whole, where their training and experience in peace-time can be utilized to the best advantage in this time of national crisis. The man skilled in processes of manufacture, if not urgently required at the present time, must turn his hand to munitions and equipment; the financier must assist in the work of safeguarding the national credit; for trained chemists there is the difficult task of preparing those substances so long supplied by those who are now our enemies; physicians, surgeons, dentists, pharmacists, and nurses must work each in their several stations; and, lastly, those who in peace-time voluntarily underwent some military training must offer their services and experience for more direct contact with the enemy.

To come to the point. Are pharmacists units that are necessary in this great national reorganization? Can they be done without? Have they been allowed or given adequate opportunities to bring into full play their trained experience and technical knowledge? If not, are pharmacists themselves somewhat to blame for not refusing to give way to influences that appear to thwart or overpower them? Has pharmacy done anything in the past to warrant her in claiming recognition as an important factor in the national life?

#### WHAT HAS PHARMACY DONE IN THE PAST?

The Government of 1868, when the demand arose that certain restrictions should be placed upon the sale of poisons, were shown by the Pharmaceutical Society that a duly trained body of men was already in existence capable of undertaking this, and as a result the Pharmacy Act, 1868, was added to the Statute Book, and for nearly half a century pharmacy has been responsible for the safe custody of and the dispensing and sale of poisons, a duty which it has performed with discretion and success. This in itself entitles it to the thanks of the Government and the community. To enable it to perform this work satisfactorily it has been necessary from time to time to extend the Poison Schedules and the scope of the examinations for the registration of those entrusted with the dispensing and sale

of poisons. Incidentally, by so doing it has advanced the training and education of the pharmacist and produced a body of men well equipped and ready to undertake sterner duties at the present time.

Pharmacy, too, has rendered material assistance in the production of successive editions of the *British Pharmacopœia*. Whether pharmacists will again form a Committee of Reference in Pharmacy or consent to play the same part in the future editions remains to be seen; but from the remarks made by the leaders of pharmacy it would appear that this is unlikely, unless more adequate representation is given to pharmacy upon the Committee which finally approves the matter for press. In a normal year we should, on the occasion of our first annual meeting after the introduction of a new edition of the *British Pharmacopœia*, have had several papers upon the work; the absence of these upon this occasion must not be taken by the compilers as evidence of its perfection, but rather as evidence of more pressing interests at the present time. One can, however, strongly urge that if the *British Pharmacopœia* is to remain a standard as between the prescriber and dispenser, another sixteen years should not be allowed to lapse until one authority or another is empowered to produce a further edition.

In respect to the National Insurance Act, 1911, it must surely be conceded that thanks to the excellent organization devised by the Pharmaceutical Society of Great Britain the pharmacists of the country have played their part extremely well, and have endeavoured to carry out their duties under this most intricate Act in a public spirit worthy of all praise. Many pharmacists undertook the work under this Act and put up with the discounting clause and its consequences in order to help forward the principle that dispensing of medicines should be done by those who have special training in this work, and one hopes that in all the delicate deliberations in which the Pharmaceutical Society has to meet the various representatives of other interests this fact is not lost sight of.

So much for the past.

#### WHAT IS PHARMACY NOW DOING,

and in what way has it responded to the nation's call at this critical time? To a very large extent, it is true—possibly for a very considerable majority—the duty of the pharmacist at the present time has been to continue to carry on business as

usual. To many, however, with previous military training, and after offering themselves perhaps for work in various capacities connected with the practice of pharmacy in the Army, the desire to take up active military work has been too strong to resist.

The pharmaceutical rolls of honour are still incomplete and can well wait for the present, but I am informed on the authority of the editor of one of our weekly journals that "It would be perfectly safe to say that about 5,000 men connected with British pharmacy and the drug-trade at home and abroad are with the Forces, and that more than 1,000, probably nearer 2,000, are connected with retail pharmacy at home." This corresponds with another editor's view, who also judges the latter figure as 2,000. This number would, I feel sure, have been immensely increased had suitable opportunities been given to pharmacists to serve in those capacities analogous to those of the medical profession, for which their special training fits them.

To digress for a moment. It has been common in past years for the occupants of this chair to deal very largely with the special events connected with pharmacy during the past year, and I cannot but feel that upon this occasion the President of the Conference should have been able to follow the various problems connected with the supply and distribution of drugs and chemicals during war-time. The tracing of these difficulties and the endeavours made to obviate them would have made an exceedingly interesting and valuable contribution at the present time, but this, I am afraid, now waits for my successor.

There are two points, however, I wish to emphasize :

(a) The necessity for an increased cultivation of medicinal plants within the Empire, as suggested in Sir Edward Evans's address at Edinburgh ;

(b) The necessity for a closer co-operation in research between chemists and physiologists for the production of new synthetic compounds or those already in use.

The Government, early in the war, saw the necessity of obtaining expert advice upon this matter of the supply of drugs and chemicals and consulted the Pharmaceutical Society. We learn, too, from the President of the Society that they were of assistance to the War Office in organizing their drug and medicine business, and we sincerely hope that the efforts that they are making to show them the advisability of employing more trained pharmacists in the collection and distribution of drugs in the Army



will be successful. Is it too much to hope that the outcome of this will be to see

### AN ARMY PHARMACEUTICAL CORPS

instituted, with a Director of Pharmaceutical Services at the head, as a separate War Office establishment? Why not? To those who have discussed this matter of the relationship of pharmacy and the Army with Continental pharmacists our position as pharmacists, or rather lack of position, has been a source of surprise, and has been put down probably to the fact that, our Army being small, there was no necessity nor place for a Pharmaceutical Department in the British War Office; but now we have a force whose dimensions, as the Secretary of State for War stated on July 9, have already reached a figure which only a short while ago would have been considered utterly unthinkable, the case is certainly different. There are two branches of the work that should be considered:

(1) The supply, collection, and storage of the necessary drugs and dressings;

(2) The provision of trained men to supervise this supply and its distribution to the various units in the base and field hospitals.

#### *1. The Supply of the Drugs and Dressings.*

The State, as we all know, are not manufacturers at the present time, and have had to rely upon wholesale manufacturers for medical and surgical supplies, and these fortunately have been able to keep things going in a remarkable manner. I cannot but feel that it might be wise for the State to have in peace-time a separate organization for the production, as well as for the collection and storage, of medical necessities capable of expansion in war-time so as to enable them to supply at once a proportion at least of the quantities required. By this means they would also have in continuous training in Army methods a body of men which in a sudden emergency could be easily augmented.

*2. The provision of trained men to act on the personnel of the supply department, the base hospitals, and the larger units in the field.*

The duties of such men would be—

*In the Central Store:* Supervision of the processes of manufacture, standardization of the various products and analyses

of those collected from other makers, and the checking of the indents received from the different hospitals.

*In the Base Hospitals :* The requisition from the central store of the various substances and appliances required in suitable quantities and containers. The distribution of these to the various wards within the hospital, having due regard to economy and prevention of waste. The careful supervision of poisonous drugs. The supervision of the dispensing of the prescriptions written by the medical staff, and full responsibility for its accuracy. The preparation of many of the simpler pharmacopœial preparations in order to avoid transport, unnecessary accumulations, and subsequent disposal at the end of the war. The responsibility for and supervision of the sterilization of dressings, instruments, and appliances. The suggesting to medical officers of alternative preparations and methods of administration, etc. The preparation of the isotonic solutions, and, in fact, all those duties which come within the province of the pharmacist at the large civilian hospitals.

*In Field Hospitals :* The duties would be as in base hospitals, only in a lesser degree, and to these might be added water-analysis and certain clinical and other analyses. The preparation of solutions to combat the effect of asphyxiating gases. To assist generally in sanitation and disinfection.

I feel that at such a time as this, when there is said to be a serious shortage of medical men, the assistance thus rendered by the trained and experienced pharmacists must result in a consequent relief of some of the duties of the medical officers, and especially of the registrars at the different base hospitals. I maintain also that this work done by pharmacists would result in greater all-round efficiency and economy.

The Army Pharmaceutical Department should also have some control in the training and examination of army compounders in co-operation with a medical officer.

### THE QUESTION OF RANK.

This is a question which for some years past has exercised the minds of qualified pharmacists and I maintain can only be considered in relation to the responsibility and importance of the work to be performed. In the duties detailed above the pharmacist would need the authority of commissioned rank, and it is this authority which is even more important than social status or rate of pay if the work is to be done effi-

ciently. The holding of a pharmaceutical qualification may not of itself be a sufficient recommendation to a commission, unless accompanied by an all-round experience in the practice of pharmacy and an ability to command.

The need for such men in the commissioned rank is not so evident at the present time, because of the self-sacrificing devotion and patriotism of many pharmacists who have enlisted and are doing much of this work with the rank of private, corporal, or at the most sergeant, and as civil dispensers. These men are learning much Army routine—the necessary forms and procedure, drill, and discipline—and when the time is ripe for reorganization it is inconceivable that their devotion to duty and the very success of their work should of itself act prejudicially to their proper recognition, but that they may be rewarded by being admitted into the commissioned ranks of His Majesty's Army.

Such, then, is my dream of the Army Pharmaceutical Service or Corps as a separate establishment of His Majesty's Army. It must be taken as a rough outline of general principles rather than a detailed scheme.

Since writing the above I have remembered that Surgeon-General Evatt before an evening meeting of the Pharmaceutical Society in November, 1906, gave an address upon this subject, describing and outlining a scheme of a pharmaceutical reserve of officers, and I would commend a perusal of this to all interested in this subject, also his paper previously contributed to the *Pharmaceutical Journal* in 1906. In the course of his address he remarked that his desire was to see—and he was doing his best towards the accomplishment of his ideal—a National Army, and he urged that when the roll of that great Army alluded to was called the pharmaceutical profession would have its proper position in it as an important factor in its efficiency. We can but echo the sentiments there expressed.

In the course of these remarks I have endeavoured to point out the advantage of national organization, so that the experts may be found out and utilized in all the branches of national service; the organizing ability of pharmacists and the past success of their work; the efforts that individual pharmacists are now making on behalf of their country; and a scheme in which the special training and experience of the pharmacist can be brought into full play and used for the public good.

We have been recently told that it is the "elementary duty

of every citizen to place the whole of his strength and resources at the disposal of his native land in its hour of need." It may be presumption upon my part to say so, but I believe that while pharmacists are willing to undertake that work which their hand finds to do, it is upon the lines I have endeavoured to indicate that the members of our calling consider they can best express their usefulness. Lord Kitchener reminded his audience at the Guildhall last week that all the reasons which led him to think in August, 1914, that this war would be a prolonged one hold good at the present time. We know also that vast numbers of men will be engaged in the fighting line and the casualty lists will be many, and heavy demands will be made upon the resources of the Medical Corps. I trust, therefore, that pharmacists will be asked to share in this work—to take their place as pharmacists in the National Army with due recognition of their qualifications, services, and powers of organization. If that demand were made I am confident that the response would be a ready one, and that pharmacists would vie with one another in enthusiasm to assist their country at this critical time. Finally, let us remember the words of Pitt:

*"What it is we have at stake, what it is we have to contend for. It is for our liberty, it is for our independence, nay for our very existence as a nation."*

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#### VOTE OF THANKS TO THE PRESIDENT

Mr. N. H. MARTIN said that before he left home he knew that the veteran senior past-President, Mr. S. R. Atkins, would not be present at the meeting, but he had hoped that the second senior past-President, Mr. Chas. Umney, would have been present and thus would have undertaken the duty which the tradition of the Conference imposes upon its oldest members. The function he spoke of was to voice the thanks of the meeting, and to voice also the wider thanks of the whole of the members of the British Pharmaceutical Conference, to the President for his address. That day they had met on a unique occasion, and he ventured to suggest that they had had a unique address. It was indeed fortunate that the President of the Pharmaceutical Conference on this occasion was a soldier. His opening remarks were those of a soldier, and so were his concluding words. There were many points in the address and many suggestions which were exceed-

ingly valuable, and when the address was printed it would prove certainly one of the most valuable which had ever appeared in the *Year-Book*. There were some suggestions in the address with regard to the Pharmacopœia, but he felt sure that before another edition of the Pharmacopœia was published pharmacy would have its proper due. Perhaps it might be that they would be enrolled as pharmaceutical units in the British Army. He could endorse from his own experience many of the remarks which had fallen as to the Government's lack of supplies. The failure to supply drugs and dressings was equally great, if not greater, than the failure to supply munitions. He had great pleasure in proposing that a hearty vote of thanks be given by the meeting to Major Peck for his address, which he felt sure would be read with very great interest by all pharmacists.

Mr. E. M. HOLMES, in seconding the vote of thanks, said that he had known Major Peck for a great many years, and he had always found that whatever Major Peck undertook he carried it through most thoroughly. What he had done so thoroughly now was only in keeping with his other good work. The address was admirable in many ways. There were a number of suggestions put forward which were well worthy of attention, and it was exceedingly appropriate because it was an address suited to the times. Though he (the speaker) had attended many Conferences during the last forty years he considered the address second to none, and one of great importance to pharmacy.

The vote was carried by acclamation.

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#### THE HON. TREASURER'S REPORT

Mr. D. LLOYD HOWARD said that the expenditure had gone on in regular lines, and the difference in the items which caused the balance of 3s. 7d. to be expanded to a balance of £84 17s. 4d. was that they had not had the expense they had had in the previous year of entertaining foreign visitors, and the expenditure on the *Year-Book* was somewhat reduced. He thought that it was a very good thing for the Conference that they started the year with a substantial balance, because although the amount received for subscriptions was very good up to date—£260 against £295 at the corresponding date last year—and subscriptions continued to come steadily in, he thought it was pos-

sible that they would want all the money they had got in order to carry on the work of the Conference adequately. He was certain that he would have the Conference with him when he expressed the opinion that it was very necessary that they should keep the *Year-Book* fully up to the standard. In the Bell and Hills' Fund they drew down their balance a little, but there was a balance remaining of £21 14s. 5d. The Research Fund remained as it was. Possibly it was too much to expect that much pharmaceutical research would go on this year, but he hoped that the younger investigators especially would remember that they had got the Research Fund, and that there was a balance remaining now of £18 12s. He hoped any one who had an idea for useful work which required rather more expensive materials than he would feel inclined to pay for out of his own pocket would remember that there was a fund of the kind at his disposal, and when it was exhausted he had very little doubt they would be able to get another together. There was every reason why that fund should be used, and not be simply left idle, as it has been during the last year.

The PRESIDENT said they had heard the Treasurer's Report, and he was sure that they were very grateful to him for his work during the past year. To his mind, the report was a very satisfactory one, and he hoped they would be able to keep the subscriptions going, so that the *Year-Book of Pharmacy* might continue its successful career.

Mr. W. L. CURRIE, in moving the adoption of the Treasurer's Report, said that he congratulated the Treasurer on the statement which he had put before them. He thought that in this abnormal year it was wonderful that he had got the balance which he had told them of. Every one knew that it entailed a considerable amount of work on the part of the Treasurer to get up a statement such as they had just heard, and the best thanks of the Conference were due to the Treasurer for the efforts which he had put forward. It was also due that he should incorporate, when moving the adoption of this report, an expression that the best thanks of the Conference be also given to the Auditors. The audit was not, perhaps, a very arduous duty, but still it was work which required to be done, and he had the greatest pleasure in moving, not only the adoption of the Treasurer's Report, but in also thanking the Treasurer for his services and the Auditors likewise.

Mr. PETER MACÉWAN said that he cordially endorsed all

that Mr. Currie had said. He thought that Mr. Howard was to be most cordially congratulated. This was a short meeting—a war meeting—so that it was desirable to come to the point; he hoped the balance would not prevent those who had not paid their subscriptions from sending them in at once, nor that it would prevent more joining the Conference during the coming year, so as to demonstrate to the whole world that although they were at war they were keeping up their end well. At all events, it was not German pharmacy that this Conference would expound in the future. He cordially seconded the resolution.

The report was adopted.

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#### REPORT OF THE EXECUTIVE COMMITTEE

Mr. R. R. BENNETT read the following report:—

The year 1914 will be for ever memorable in the world's history, and, since the Executive presented its Fifty-first Annual Report at Chester pharmacists have had to help in the burden which every class of the community has been called upon to bear.

It will be fresh in the minds of every one that at the Chester meeting the pharmacists of Scarborough offered the members of the Conference an invitation to hold their fifty-second meeting at Scarborough, and the Mayor of Scarborough supported this invitation by sending a personal letter to every member of the Conference. The outbreak of war has altered all pre-arranged plans, and the Hon. Local Secretary of the Scarborough Committee communicated to a meeting of the Executive in January last that it was the unanimous feeling of the Scarborough Committee that it would not be desirable to hold the annual meeting in Scarborough this year. The Executive accordingly decided to postpone the proposed visit, and arranged to hold the annual meeting in London without the usual social functions. At a subsequent meeting of the Executive it was decided that the annual meeting of members should be restricted to purely formal business, and that no meetings of the Science and Practice Sections should be held, but that all accepted contributions to these two sections should be printed in full in the *Year-Book of Pharmacy* for 1915.

The Practice Section Sub-Committee, consisting of the President and Messrs. T. O. Barlow, H. Finnemore, E. F. Harrison, F. W. Gamble, T. Stephenson, G. Whitfield, H. Wyatt, with

R. R. Bennett as convener, has been chiefly concerned with the drafting of a set of rules for the use of medical practitioners complementary to the code of rules which, as an outcome of Mr. Peck's paper on "Uniformity in Dispensing Abnormal Prescriptions," were recommended for adoption by all pharmacists at the meeting of the Practice Section of the Conference at Chester.

The rules drafted by the Practice Section Sub-Committee were submitted to a meeting of the Executive, and were forwarded to the British Medical Association with an invitation to meet representatives of the Conference in order that the suggested rules might be discussed. The President and Messrs. T. O. Barlow, F. W. Gamble, and one of the Secretaries were appointed to represent the Conference at any joint discussion that might ensue. In due course the Science Committee of the British Medical Association appointed Professor A. R. Cushny, F.R.S., and Professor Ralph Stockman as representatives of the British Medical Association to confer with representatives of the Conference. The joint meeting was held last month, the rules were freely discussed, and, after amendment, were re-drafted and are now in the hands of the Science Committee of the British Medical Association for further discussion.

The Executive wish to express their appreciation of the sympathetic and helpful manner in which the representatives of the British Medical Association have dealt with the suggested rules, and they confidently believe that the friendly feeling which has always existed between the British Medical Association and the British Pharmaceutical Conference will be further strengthened by the matter in hand.

The Research Sub-Committee, consisting of the President and Messrs. R. R. Bennett, H. Deane, F. W. Gamble, C. H. Hampshire, E. F. Harrison, C. A. Hill, D. Hooper, with H. Finne-more as convener, met early in the year, but it was the unanimous feeling of the meeting that the time was not opportune for the initiation of new research work.

The Conference Development Sub-Committee, consisting of the President and Messrs. T. O. Barlow, R. R. Bennett, H. Finne-more, E. F. Harrison, E. M. Holmes, D. Lloyd Howard, and G. Whitfield, also met early in the year to consider and make recommendations in regard to measures that can be taken to extend the usefulness of the Conference. The report of this Sub-Committee was submitted to the Executive, but it was



agreed that the discussion upon the report should be postponed for the present.

Every effort has been made to maintain the high standard of the *Year-Book of Pharmacy*, and the Executive wish again to record their appreciation of the services of the Editor of the abstracts, Mr. J. O. Braithwaite. Despite the unsettled period which has elapsed since the publication of the last *Year-Book*, the Executive anticipate that the high standard of the abstracts in the *Year-Book* for 1915 will be maintained.

The Executive record with regret the deaths of the following members of the Conference since its last meeting:—Sir Arthur Church, Kew; Messrs. F. W. Ashton, London; R. Brodie, Glasgow; J. Hallaway, Carlisle; T. Kay, Stockport; J. Nesbit, Portobello; W. Ransom, Hitchin; W. Saunders, Ontario; W. G. Strongitharm, Dublin; J. A. Thomas, Cheltenham; R. L. Whigham, London.

The Executive desire to thank the Council of the Pharmaceutical Society for making provision for the meetings of the Executive during the year, and also for providing accommodation for the present annual meeting.

Mr. W. G. CROSS said that it gave him extreme pleasure to rise to propose the adoption of the Annual Report. With regard to the work of the various committees, naturally during such a year as had passed they had dealt largely with matters which had had to be postponed as incomplete. But he believed that although pharmaceutical research had been postponed for a time they could look back to this strenuous time as one which had afforded much matter for research. One thing was particularly evident, namely, the need of pharmacy and pharmacy methods in the various departments of State, and he trusted that when the time came for reorganizing the various departments of State that pharmacy, which had been kept alive and fostered by the Conference, would have due place.

Mr. E. H. FARR said that he had very much pleasure in seconding the motion for the adoption of the report. The Executive Committee during the past year had had very largely to mark time. With reference to the absence of scientific papers on this occasion, naturally all the members who had not joined the Forces had taken some part in warlike preparations and were overwhelmed with work, and research was absolutely out of the question. There was one point in connexion with the report which all would be very pleased to note, and that was the co-operation with the British Medical Association.

The PRESIDENT said that he would like to emphasize the cordiality with which representatives of the Conference were received by the two representatives of the Science Committee of the British Medical Association, Professor A. R. Cushny and Professor Ralph Stockman. There had been a most useful two hours' discussion, and he felt sure that pharmacists would be rather pleased with what had taken place during the meeting.

The report was adopted.

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• ELECTION OF OFFICERS FOR 1915-1916

On the motion of Mr. J. P. Gilmour, seconded by Mr. F. W. Gamble, the following officers were elected for 1915-1916:—President—Major E. S. Peck; Acting-President—J. C. Umney; Vice-Presidents—E. F. Harrison, D. M. Watson, Edmund White, G. Whitfield; Hon. Treasurer, D. Lloyd Howard; Hon. General Secretaries—H. Finnemore and R. R. Bennett; Hon. Local Secretary—H. Humphreys Jones; Other Members of the Executive—T. O. Barlow, H. Deane, F. W. Gamble, C. H. Hampshire, C. A. Hill, D. Hooper, W. F. J. Shephard, T. Stephenson, Harold Wyatt; Auditors—I. Bourdas, W. F. Gulliver.

Mr. J. P. GILMOUR said that he had the honour to move the election of the office-bearers nominated by the Executive Committee of the Conference. He wished to pay a tribute to the work which the President, Major Peck, had done during the past year. He had fulfilled all the duties he was called upon to fulfil in his characteristically efficient way. There was on this occasion a new officer on the list, that of "Acting-President." While Major Peck had found it barely possible for him to get through the work during the past year, it would be physically impossible for him to do it in the coming year. In these circumstances it had been found necessary to nominate an Acting-President. The name selected, Mr. J. C. Umney, was a sufficient recommendation. It was another example of that disinterested sense of public duty and what the old Romans called "citizenship" which had always distinguished Mr. Umney.

Mr. F. W. GAMBLE said that it was merely a formal duty for him to second the proposal for the election of the list of officers. He thought that the solution of the difficulty that the Executive were recommending was the best solution that could be made. Major Peck had not had a chance that year. And of all the

hard workers in the Conference it was the President whom he thought should be retained for two, or perhaps even for three, years. The method of giving him assistance by appointing an Acting-President was an excellent one; and it was gratifying to all that Mr. Umney was able so adequately to fill the breach. It might, perhaps, have escaped the notice of some that Major Peck had recently received a promotion, which had been the reward for the tremendous work which he had put into his military duties. And there was one thing which had not been mentioned, and that was that, Major Peck might not be with them during the whole year, so he would therefore take that opportunity of saying how they all hoped he would have every success and honour.

Mr. N. H. MARTIN put the resolution to the meeting, and it was carried by acclamation.

The PRESIDENT said that he was not at all sure that the Conference had done the right thing in making him President for another year. But his duties would, he was sure, be admirably performed by the Acting-President in his absence. He would like publicly to thank Mr. Umney for so kindly coming forward to take the position of Acting-President.

Mr. J. C. UMNEY said that he would do his very best to act in the President's absence.

The PRESIDENT said that he wished to make one further remark. The work of the Conference during the last year had been largely carried on by the two Secretaries—Mr. Finnemore and Mr. Bennett—and he wanted to propose a vote of thanks to them. He was sorry Mr. Finnemore was not with them, but he believed that he was hard at work training himself for taking up more active work in military matters later on. He was a member of the Inns of Court Officers' Training Corps, and he wished him all success.

Mr. C. A. HILL said that he seconded the special vote of thanks with pleasure.

Mr. R. R. BENNETT said that he was sure it was a great disappointment to Mr. Finnemore that he could not be present. Speaking for himself, it had always been a very great pleasure to do all that he could for the Conference.

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#### INVITATION TO LIVERPOOL

Mr. H. HUMPHREYS JONES said that it gave him the greatest possible pleasure to rise to ask the Conference to visit Liver-

pool in 1916. Two years ago he had written to Mr. Finnemore asking him for the rotation of the Conference last year, but he was told Chester had spoken for it, and, in addition, that Scarborough had invited it for 1915. Mr. Finnemore had suggested at that time that Liverpool should hold itself in readiness to follow their friends at Scarborough. There was no occasion for him to go into the reasons why the arrangements to visit Scarborough had been frustrated, but he understood that it was the intention of their friends in Scarborough to have the Conference there when things became normal. It was his pleasure to ask those present—and through them all the members of the Conference—to visit Liverpool in 1916, with the proviso, which he was sure they would consider reasonable, that the Liverpool pharmacists should have at least six months in which to make their arrangements. That was to say, if the Conference decided to hold its meeting in Liverpool next year, they should know not later than January. He was sure that all would realize that even with six months' notice the Liverpool pharmacists would be under a considerable disadvantage with regard to the making of proper arrangements, and he hoped the Conference would not compare them with the arrangements which have been made under different conditions. But, whether the arrangements were elaborate or not, they would receive from Liverpool pharmacists a very warm welcome. It was twenty years since the previous visit of the Conference to Liverpool, and that Conference was notable in many respects. In the first place, the members were officially welcomed by one of the most famous of Lord Mayors, namely, the Earl of Derby, the father of the present Earl. The meeting was presided over by one of the greatest English pharmacists who ever lived. He referred to Mr. Martindale. And it was the only occasion on which the Conference had been addressed by that great English statesman, Mr. Gladstone. Dr. Symes was Chairman of the Executive Committee and, unlike other notabilities, Dr. Symes was still with them, and was as nimble and as energetic as ever. He read a letter from Mr. W. P. Evans heartily supporting the invitation to Liverpool.

Mr. H. WYATT said that he seconded the invitation with extreme pleasure. He did not regard the entertaining of the Conference in Liverpool as a burden; it was an honour to Liverpool, and would give the greatest possible pleasure to the pharmacists of that city.

Mr. J. C. UMNEY, in the name of the Conference, expressed warm thanks to Liverpool for the invitation.

Mr. G. WHITFIELD said that he hoped that when things were different nationally, Scarborough would be able to renew her invitation to the Conference, and secure its acceptance. As a member of the Executive and as representing Scarborough, he was grateful to Liverpool for having filled the breach. They were doing it at short notice ; but Liverpool was a great city, and no doubt they would do things well.

The invitation was accepted with acclamation.

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#### VOTE OF THANKS TO THE PHARMACEUTICAL SOCIETY

Mr. F. RANSOM proposed that hearty thanks be accorded to the Council of the Pharmaceutical Society for the use of their premises for the purpose of the meeting. The Conference was really the offspring of the Society, and had been glad of the association from the time of its birth.

Mr. T. O. BARLOW seconded in an appreciative speech, and the vote was carried.

Mr. P. F. ROWSELL acknowledged the vote, taking the occasion to pay a tribute to the able presidency of Mr. Edmund White over the Society.

The PRESIDENT announced the receipt of a science paper from Messrs. G. D. Elsdon and Herbert Hawley, which would appear in the *Year-Book* ; and the meeting thanked those gentlemen by resolution. The meeting also approved the suggestion of the President to send the following telegram to Mr. S. R. Atkins, of Salisbury, the Senior Past-President :—"S. R. Atkins, J.P., Salisbury. To the veteran pharmacist, our senior Past President, the Conference assembled at Bloomsbury Square send hearty congratulations and good wishes.—PRESIDENT."

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At the termination of the meeting most of those present lunched together in the Gordon Room at the Holborn Restaurant. There were no speeches, and the only toast was that of "The King," proposed from the chair.

## PAPER COMMUNICATED TO THE SCIENCE SECTION.

THE EXAMINATION OF PULVIS RHEI COMPOSITUS  
(GREGORY'S POWDER)By G. D. ELSDON, B.Sc., F.I.C., and HERBERT HAWLEY, M.Sc.,  
F.I.C.

Samples of Gregory's Powder (Pulvis Rhei Compositus B.P.) are frequently submitted for analysis under the Sale of Food and Drugs Acts, and it is, therefore, a matter of some importance to be able to examine them rapidly with sufficient accuracy.

According to the British Pharmacopœia of 1914, Gregory's Powder consists of light magnesia ( $MgO$ ), 66 (the new Pharmacopœia orders the light variety, although either is allowed in the 1898); rhubarb root, in powder, 22; ginger, in powder, 12. This is slightly different in composition to the formula of 1898 (magnesia, 6; rhubarb, 2; ginger, 1), but the difference is not sufficient to invalidate the results given in this paper which have been obtained for the 1898 formula. The most likely adulterations are the substitution of magnesium carbonate for oxide (in fact, a formula for "improved" Gregory's Powder, containing B.P. magnesium carbonate in place of oxide, is contained in the British Pharmaceutical Codex) and careless dispensing. A powder left long, badly stored, or made from old materials will also contain a large percentage of carbon dioxide derived from the atmosphere, gas fumes, etc.

*The Determination of Moisture, Ash, and Magnesia.*—The moisture (as represented by the loss at  $100^{\circ}C$ . when heated for four hours in a 3-inch flat-bottomed metal dish in the water oven) and ash may well be determined on the same quantity of 1 Gm. The loss at  $100^{\circ}C$ . will not be entirely due to moisture, but the excess will be more or less compensated by the absorption of carbon dioxide; in any case, however, figures obtained under similar conditions are strictly comparable. Fifteen commercial samples examined by the authors have lost between 2.1 and 5.0 per cent. Only two of these, however, lost more than 4.0 per cent., and this is suggested as a reasonable limit; 3 samples of B.P. magnesium carbonate have lost less than 0.5 per cent.; 10 samples of ginger an average of 10.5 per cent., and 10 samples of rhubarb an average of 9.5 per cent.

The figure obtained for the ash will be due to magnesium

oxide, together with the ash from the ginger and rhubarb. The calculated ash, using B.P. MgO and allowing the average figures 10.0 per cent. for rhubarb (samples having high ash figures are not usually used for grinding purposes), and 5.0 per cent. for ginger, is 67.5 per cent. Fifteen commercial samples have given figures varying from 57.1 to 72.5 per cent.

The alkalinity of the ash can then be determined by titrating with N/1 hydrochloric acid to methyl orange. The figure so obtained calculated as MgO is usually about 4.5 per cent. less than the ash figure. This difference is, of course, due to the presence of calcium oxide and to the non-alkaline constituents of the ash of ginger and rhubarb—as would be supposed it is somewhat variable. The neutral liquid may be tested qualitatively, and, if required, the magnesium may be determined gravimetrically on an aliquot portion.

*Determination of Carbon Dioxide.*—The method first tried for this determination was that of Paul and Cownley (*P.J.*, 61, 389), which is as follows :—0.5 Gm. (or less if much carbonate is present) of the powder is rubbed down with 3 c.c. of water and washed into the cup of a nitrometer standing over mercury, with two further quantities of 1 c.c. of water and the whole allowed to enter the graduated tube; 5 c.c. of concentrated hydrochloric acid are then added, the whole well shaken, and the volume read. The volume of the gas obtained is corrected for temperature, pressure and solubility. They found the solubility of carbon dioxide in 10 c.c. of the liquid to be 1 c.c. at 60°F., and 758 mm., but they make no mention of the method by which this figure was obtained.

It soon became apparent, however, that this method was giving erroneous results, and it was found by preliminary experiments that a correction of about 8.0 c.c. was necessary at 60°F. and 760 mm. in place of the 1 c.c. recommended by Paul and Cownley. Further experiments, however, revealed the fact that the solubility was largely dependent upon the amount of magnesium oxide that had been dissolved. The solubility of the carbon dioxide was then determined in acid solutions of various strengths.

A solution of magnesium oxide in  $\frac{N}{5}$  hydrochloric acid was prepared, and this solution was mixed with  $\frac{N}{5}$  hydrochloric acid in varying proportions, to give liquids containing varying

amounts of magnesium chloride in  $\frac{N}{5}$  hydrochloric acid. Ten c.c. of this liquid were used for each determination, the carbon dioxide being prepared from marble and hydrochloric acid.

The nitrometer was completely filled with carbon dioxide by passing the gas through from the top for some time, allowing it to bubble through the mercury. The mercury was then allowed to rise until a convenient volume remained in the nitrometer. Ten c.c. of the acid liquid were then introduced and the tube shaken at intervals, until the volume of carbon dioxide was constant, and the decrease in volume noted.

The results are given in the following table :—

Grams MgO in 10 c.c. $\frac{N}{5}$ HCl.						C.c. of CO <sub>2</sub> dissolved at 65°C. by 10 c.c.					
0	.	.	.	.	.	.	.	.	.	.	7.5
0.15	.	.	.	.	.	.	.	.	.	.	6.3
0.25	.	.	.	.	.	.	.	.	.	.	6.1
0.35	.	.	.	.	.	.	.	.	.	.	5.6
0.5	.	.	.	.	.	.	.	.	.	.	5.1

The determination of carbon dioxide can thus be carried out by the method of Paul and Cownley, provided that the correction to be applied be taken from the above table. An amount of powder should be taken such as will give from 2 to 5 c.c. of undissolved gas, but it is not convenient to work with quantities greatly exceeding 0.5 Gm. Working on 0.5 Gm., the corrected number of c.c. of carbon dioxide obtained at 65° and 760 mm., multiplied by 0.364, gives the percentage of carbon dioxide in the sample.

No samples have been met with that could be classed as genuine which have contained more than 5 per cent. of carbon dioxide, whilst in most cases the amount has been less than 3 per cent. The question of the amount of carbon dioxide admissible is further discussed below.

*The Determination of Rhubarb and Ginger.*—The combined amounts of ginger and rhubarb can be obtained roughly from a consideration of the ash, and the amount of magnesium. A confirmation can be obtained in the following manner. The aqueous extract of the powder is obtained by treating 1.2 Gm. with 60 c.c. of cold water for 12 hours, filtering, and evaporating 50 c.c. (=1 Gm. of sample) to dryness in a flat-bottomed dish, and drying until constant in weight. One Gm. of the



powder is treated with 70 c.c. acetic acid (of about  $\frac{N}{2}$  strength) in a conical flask overnight, filtered through a Gooch crucible or a tared filter paper, and the residue washed once with water and dried in the steam oven. The weight of residue obtained, together with the aqueous extract, after adding 10.0 per cent. of the total weight for the average moisture in ginger and rhubarb (this figure is very constant), gives the amount of rhubarb and ginger in the sample. This depends on the fact that dilute acetic acid extracts nearly the same amount from rhubarb and ginger as does water, but dissolves in addition the magnesium oxide.

The relative amounts of rhubarb and ginger are not so easy to determine, but a very fair approximation may be obtained by the following process. The aqueous extract has already been obtained, and the alcoholic extract is now obtained in a similar manner, using industrial methylated spirit 64°O.P. From the percentage extracts of the Gregory's Powder, the percentage extracts of the combined rhubarb and ginger mixture contained therein are obtained. The extracts of ginger are remarkably constant, 30 samples of ginger, in the authors' hands, having given an average of 13.3 per cent. aqueous extract and 5.8 per cent. alcoholic extract. The extracts of rhubarb are, however, unfortunately not so constant, 15 samples giving from 44.0 to 30.0 per cent. aqueous extract and from 36.4 to 28.8 per cent. alcoholic extract. Further results are given by Brewis and Deane (*Y.B.P.*, 1913, 524), who, however, use 50 per cent. by volume alcohol. However, for calculation purposes, taking into consideration the kind of sample that is usually used for grinding, an average of 38.0 per cent. may be taken for the aqueous extract, and 33.0 per cent. for the alcoholic extract.

Taking 38 and 13 as the percentage aqueous extracts of rhubarb and ginger respectively, and 33 and 6 as the percentage alcoholic extracts respectively, and knowing the aqueous and alcoholic extracts of the mixture, it is, of course, possible to calculate the composition of the mixture from either of these figures; the latter is, however, much more accurate.

The following example may make this clear. A Gregory's Powder had an aqueous extract of 11.6, whilst 20.1 per cent. was insoluble in  $\frac{N}{2}$  acetic acid. The total amount of rhubarb

and ginger is therefore  $11.6 + 20.1 + \left( \frac{11.6 + 20.1}{10} \right) = 34.9$  per cent. The alcoholic extract was 7.8 per cent. Calculated, therefore, on the alcoholic extract, the proportion of rhubarb and ginger was 62 : 38.

*The Effect of Exposing Gregory's Powder to the Atmosphere.*—A number of results were given in Paul and Cowley's paper, showing the effect on the composition of leaving magnesium oxide exposed to the atmosphere for varying lengths of time, and they conclude that an allowance of 5 per cent. of B.P. magnesium carbonate in magnesium oxide is ample. The method of exposure was not, however, given, and no details as to the presence or absence of gas fumes, ventilation, etc., were stated. Similarly, experiments have been carried out with Gregory's Powder. A quantity of powder was spread out in a thin layer and placed in a well ventilated, closed, first floor outside window box in one of the busiest thoroughfares of a manufacturing city. The results are given below.

Exposure commenced on July 22, 1914.

Total given up to July 31	.	.	.	2.22 per cent.*
" " " August 31	.	.	.	6.25 "
" " " September 29	.	.	.	6.25 " †
" " " November 4	.	.	.	12.23 "

Analysis before exposure.			Analysis after exposure.	
Loss at 100°C.	.	3.8		7.2
Ash	.	67.3		61.6
Alkalinity of ash as MgO	.	62.7		57.8
Aqueous extract	.	14.1		13.1
Carbon dioxide	.	2.2		3.5

Quantities of B.P. magnesium oxide and B.P. magnesium carbonate were exposed in a similar manner, the oxide being ignited and the carbonate being dried at 100°C. at the commencement of the experiment. Commenced November 20, 1914.

		Mg. Ox.	Mg. Carb.
Total gain in weight to December 22, 1914		15.6	2.6
" " " January 23, 1915	.	18.1	3.0
" " " February 23, 1915	.	19.6	3.6

The final products were examined with the following results :—

\* On exposure the powder becomes much pinker—this might conceivably be useful in detecting exposure.

† There was thus no change in the month, which was a dry one.

	Exposed Mg. Ox.	Exposed Mg. Carb.
a. Carbon dioxide . . . . .	7.8 per cent.	32 per cent.
b. Ash . . . . .	86.0 "	44.0 "
c. Alkalinity as MgO . . . . .	40.6 "	20.3 "
d. Loss at 100°C. . . . .	1.5 "	2.5 "
e. Portion due to increase of exposure . . . . .	17.3 "	3.5 "
f. Portion due to dust and fixed H <sub>2</sub> O . . . . .	8.0 (c - a - d)	1.0 (e - d)

From these and similar results, and from our general experience, we consider that Gregory's Powder should not contain more than about 3 per cent. of carbon dioxide, and that quantities of more than 5 per cent. certainly constitute adulteration.

We append complete analyses of a number of samples of Gregory's Powder bought under the Sale of Food and Drugs Acts.

Moisture per cent.	Ash per cent.	Alkalinity of Ash as MgO.	CO <sub>2</sub> per cent.	Cold Water Extract.	Insoluble in $\frac{N}{2}$ HA
4.1	47.2	44.1	14.3	—	—
4.9	49.4	44.9	14.8	12.1	18.6
2.9	68.2	63.4	2.9	—	—
2.4	72.5	66.3	2.9	13.3	17.5
3.5	67.6	63.0	2.9	11.6	20.1
3.0	67.0	62.2	3.2	11.1	19.7
3.1	63.6	58.4	3.2	—	—
4.3	58.0	53.8	7.0	—	—
3.4	64.9	59.6	3.3	—	—
3.1	57.1	52.3	3.0	11.6	21.8
2.1	70.0	64.9	3.0	—	—
2.3	69.1	64.2	2.9	—	—
2.6	68.1	63.6	2.9	—	—
4.0	30.6	27.1	21.6	—	—
3.9	44.2	39.0	13.4	12.6	20.8
4.8	30.6	27.4	23.0	11.7	20.0
2.9	70.9	67.2	2.9	11.0	17.2
5.0	60.8	56.4	5.2	11.9	18.1
3.4	65.8	61.8	Small	12.2	20.2
3.1	67.1	63.6	"	12.6	20.1
3.4	66.7	62.7	"	12.1	19.9

*Municipal Laboratory :*  
141, REGENT ROAD,  
SALFORD.

*Analytical Department :*  
44, BROAD STREET,  
BIRMINGHAM.



# BRITISH PHARMAC

## RECEIPTS AND EXPENDITURE FO

**Dr.**

1914.		£	s.	d.	£	s.	d.
Jan. 1.	To Balance from last year . . .				0	3	7
Dec. 31.	„ Members' Subscriptions received by Secretaries .	350	16	9			
	„ „ „ paid to Bankers	17	13	0			
		<hr/>			368	9	9
	„ Amount received for Reprints of Conference Papers . . .	4	4	0			
	„ Sale of Year Book by Publishers .	17	16	0			
	„ „ „ Secretaries .	2	15	0			
		<hr/>			24	15	0
	„ Advertisements in Year Book. . .				87	5	0
	„ Bank Interest on Deposit . . .				1	9	6
		<hr/>			<u>£482</u>	<u>2</u>	<u>10</u>

### LIABILITIES.

Butler & Tanner . . . . .	193	1	11			
T. Stephenson . . . . .	10	0	0			
Society of Public Analysts . . . . .	2	2	0			
Bell & Hills' Fund . . . . .	21	14	5			
	<hr/>			226	18	4
Balance . . . . .				84	17	4
	<hr/>			<u>311</u>	<u>15</u>	<u>8</u>

### BELL AND HILLS' FUND.

#### RECEIPTS AND EXPENDITURE FOR YEAR 1914.

1914.		£	s.	d.	£	s.	d.
Jan. 1.	TO BALANCE IN HAND . . . . .	23	3	5			
	„ DIVIDEND ON CONSOLS . . . . .	8	9	1			
		<hr/>					
	Cr. . . . .	31	12	6			
	By Gratton's a/c for Books . . . . .	9	18	1			
		<hr/>			<u>£21</u>	<u>14</u>	<u>5</u>

**ASSETS :—**

£360 Consolidated 2½% Stock and above Balance.

# ICAL CONFERENCE,

1 YEAR ENDED 31st DECEMBER, 1914.

Cr.

1914.		£	s.	d.	£	s.	d.
Dec. 31.	By EXPENSES OF YEAR BOOK (1914) :—						
	Printing, Publishing and Binding	176	16	2			
	T. Stephenson . . . . .	10	0	0			
	Posting and Distributing . . . .	16	5	9			
					203	1	11
	„ Commission on Advertisements (Churchill) . . . . .	18	16	7			
	„ Advertisements in Lists and Post- age (Churchill) . . . . .	1	3	6			
					20	0	1
	„ Editor's Salary . . . . .				75	0	0
	„ Secretarial Expenses (Assistant Secretary) . . . . .	35	0	0			
	„ „ „ Annual Meeting . . . . .	1	14	6			
					30	14	6
	„ Postage, etc. (Secretaries) £11 17s. ; (Editor) 17s. 9d. . . . .	12	14	9			
	„ Petty Cash Sundries . . . . .	5	5	7			
					18	0	4
	„ PRINTING, STATIONERY, ETC. :—						
	„ W. Straker . . . . .	2	13	6			
	„ Ash & Co. . . . .	19	19	0			
	„ Heffer's Printing Works . . . .	1	16	3			
	„ Miss Huggett (Typing) . . . . .	4	4	0			
	„ Misses Bowring . . . . .	1	13	1			
	„ Heywood & Co. (Reprints <i>Pharma- ceutical Journal</i> ) . . . . .	3	1	0			
					33	6	10
	„ Foreign Journals for Editor . . .				2	17	0
	„ Subscription American Chemical Society . . . . .				2	12	1
	„ Room for Conference (Grosvenor Hotel)				10		6
	„ Subscription and Entrance Fee Society of Public Analysts . . . .				2	2	0
	„ Bank Charge (Scotch cheque). . .						3
	„ Balance . . . . .				84	17	4
					£482	2	10

## ASSETS.

Cash at Bank . . . . .	226	8	6
„ in hands of Hon. Secretaries . .	9	9	2
Due from J. & A. Churchill . . . .	75	18	0
			311 15 8
			£311 15 8

## RESEARCH FUND.

1914.		£	s.	d.
Jan. 1-Dec. 31.	To Balance in hand . . . . .	18	12	0
	(No receipts or expenditure during 1914).			
		£18	12	0.

Examined and found correct, and signed on behalf of the Auditors,

I. BOURDAS.



## HONORARY MEMBERS

- BAKER, R. T., F.L.S., Technological Museum, Harris Street, Sydney, N.S.W.  
BOURQUELOT, Prof. Em., Journal de Pharmacie et de Chimie, Paris.  
FOURNEAU, Mons., Chef de Service à l'Institut Pasteur, 25, Rue Dutot, Paris.  
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Australia.

Browncombe, W. J., Bridge Road, Richmond, Melbourne.  
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Champion, G. A., "Haraldine," Cholmsford Road, Durban,  
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Cowell, S. G., c/o Maw, Son & Sons, 248, Queen Street, Brisbane.  
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Dey, Notendra Lall, 4, Beadon Street, Calcutta, India.

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 Ryan, F. G., c/o Parke Davis & Co., Detroit, Mich., U.S.A.

Samuel, J. B., Mussoorie, India (Year-Book and Letters to  
 A. Lawrie & Co., 14, St. Mary Axe, E.C.).  
 Say, S. V. B., Bonalla, Victoria.  
 Scammell, L. R., Adelaide (c/o F. H. Faulding & Co., 54,  
 Great Tower Street, E.C.).  
 Simmonds, F. W., Toowong, Brisbane, Queensland, Australia.  
 Sinha, B. C., 147, Baranasi Ghose's St., Calcutta, India.  
 Smith, F. A. Upsher, 2002, Inglehart St., St. Paul, Minnesota,  
 U.S.A.  
 Speechly, E., Kurachi, Scindo, India (Year-Book to Maw, Son &  
 Sons, 11, Aldersgate Street, E.C.).  
 Squire, F. R., San Remo, Italy.  
 Stevens, H. F., 140, Worcester Street, Christchurch, N.Z.  
 Stewart, A. M., 2, Bishop's Street, Penang.

Taylor, H. A., Government Civil Hospital, Hong Kong.  
 Thomas, H., Nundah, Brisbane, Queensland.  
 Thomas, H. W., 9, Dalhousie Square, Calcutta.

Wadsworth, A., Sydney, N.S.W., Australia.  
 Walker, F. G. C., F.I.C., Municipal Health Dept., Shanghai.  
 Walker, J. A., Northcote, Australia.  
 Walsh, A., Adderley Street, Cape Town (Year-Book and  
 Letters to Lennon, Ltd., 54, Queen Elizabeth Street, S.E.).  
 Wardleworth, Theo. H., F.L.S., c/o National Drug and Chemical  
 Co. of Canada, Montreal.  
 Watkins, George, 206, Queen Street, Brisbane, Queensland.  
 Watson, Edwin L., c/o D. Waldie & Co., Konnagar, E.I.R.,  
 Calcutta.  
 Wheeler, F., Grant Street, Alexandra, Victoria.  
 Wilkinson, R., Dunedin, New Zealand.  
 Woolecott, J. N., Warracknabeal, Victoria.

## HOME MEMBERS

- Abraham, Alfred C., F.I.C., F.C.S., 87, Bold Street, Liverpool.  
 Abraham, T. F., 87, Bold Street, Liverpool.  
 Aitken, Robt., 67, High Street, Dunbar.  
 Aitken, R., 73, Princes Street, Edinburgh.  
 Alcock, F. H., F.I.C., F.C.S., 5, King Alfred's Place, Birmingham.  
 Alder, Stanley Frank, 35, Granley Street, Liverpool.  
 Alexandor, Wm., 57, Low Street, Banff.  
 Allen, C. B., 20, High Road, Kilburn, N.W.  
 Allen, Charles T., 14, Honiton Road, Kilburn, N.W.  
 Allen, Edward R., 7, Cowper Street, Finsbury, E.C.  
 Allen, E. W., 7, Cowper St., Finsbury, E.C.  
 Allen, G. Stafford, Long Melford.  
 Allen, K. C., 7, Cowper Street, Finsbury, E.C.  
 Allen, W. H., 20, High Road, Kilburn, N.W.  
 Allman, J. D., 23, Kenilworth Road, Ealing, W.  
 Anderson, D., 21, Broadway Parade, Crouch End, N.  
 Anderson, James, 70-74, Commercial Street, Dundee.  
 Andrews, Fredk., 34, Leinster Terrace, Lancaster Gate, W.  
 Antcliffe, Herbert, The Beeches, Barnsley Road, Sheffield.  
 Appleton, J. T., The Walkley Pharmacy, Sheffield.  
 Arnaud, F. W. F., F.I.C., County Analyst's Laboratory, Sessions House, Maidstone.  
 Arnfield, H., F.C.S., Peak Lodge, Buxton Road, Stockport.  
 Arnfield, J. C., 7-9, Lower Hillgate, Stockport.  
 Arnfield, T. O., 7-9 Lower Hillgate, Stockport.  
 Arnold, H. R., c/o Burgoyne Burbidges, East Ham, E.  
 Arnold, S., 2, King's Road, Southsea.  
 Arrowsmith, A. R., "Dorchester," Wontner Road, Upper Tooting Park, S.W.  
 Ashcroft, A. W., 112, Aigburth Road, Liverpool.  
 Ashmore, W. Hopkins, M.P.S.I., 21, Dawson Street, Dublin.  
 Ashton, C. S., 46, Dyke Road, Brighton.  
 Ashton, H. M., 63, Sankey Street, Warrington.  
 Aston, W., 27, Montague Street, Worthing.  
 Atkins, E. A., 71, East Hill, Wandsworth, S.W.  
 Atkins, S. R., J.P., The Mount, Elm Grove, Salisbury.  
 Atkins, W. R., Market Place, Salisbury.  
 Atkinson, A. Proctor, 143, Farringdon Road, E.C.  
 Atkinson, J. G., 27, Lunham Road, Upper Norwood, S.E.  
 Attenburrow, James, 1, High Street, Melton Mowbray.  
 Aukland, W. H., 96 Camden Rd., N.W.  
 Backhouse, H. N., 76, New Bond Street, W.  
 Bain, John, "Bruntsfield," Bridge of Allan, N.B.  
 Bailey, A. E., 64 High St., Highgate, N.  
 Baker, Cyril H., Cosham, Hants.  
 Baker, H. J., 239, Elgin Avenue, Maida Vale, W.  
 Baker, T., 35, Cranbrook Street, Oldham.  
 Balcombe, J., Suffolk Parade, Cheltenham.  
 Ball, A. W., 179, Queen Victoria Street, E.C.  
 Ballantyne, W. M., 26, Clyde Street, Edinburgh.

- Balmforth, A., 5, Grosvenor Road, Whalley Range, Manchester.  
 Bannister, W., J.P., "Dunloo," Bramley Hill, Croydon.  
 Barclay, Sir Thomas, 19, Lower Priory, Birmingham.  
 Barclay, Thomas, New Charford Mills, Saltley, Birmingham.  
 Barfoot, J. R. D., 69, West Bars, Chesterfield.  
 Barford, H. W., 34, Union Street, Ryde, Isle of Wight.  
 Barlow, Alfred H., Werneth Road, Woodley, near Stockport.  
 Barlow, T. O., 2, Palmerston Road, Southsea.  
 Barnes, Ivor P., 205, Knightsbridge, S.W.  
 Barr, A. S., 38, Berry Street, Liverpool.  
 Bascombe, F., F.I.C., 17, St. Saviour's Road, Brixton Hill, S.W.  
 Basker, J. A., F.C.S., 17, Fore Street, Bridgwater.  
 Bately S. B., 682, High Road, Tottenham, N.  
 Bates, F. W., 178, Chorlton Road, Manchester.  
 Bates, W., 50, Oxford Street, Southampton.  
 Baxter G., 11, Polworth Place, Edinburgh.  
 Baxter, John, Ballymoney.  
 Baxter, Sir W. J., J.P., M.C.P.S.I., Church Street, Coloraine.  
 Bayley, Cornelius, High Street, Uppingham.  
 Bayne, C. P., Falconer, 9, Goldenacre Terrace, Edinburgh.  
 Bayne, Thomas, Blandfield Chemical Works, Wheatfield Road, Edinburgh.  
 Beacock, J. H., 20, Upperhead Row, Leeds.  
 Bell, E. Wightman, F.C.S., County Agricultural Laboratory, Spalding.  
 Bell, W. A., The Strand, Southsea.  
 Bennet, D. S., Cahirciveen, Ireland.  
 Bennett, C. T., B.Sc., F.I.C., F.C.S., 48, Southwark Street, S.E.  
 Bennett O. E., Royal Waterloo Hospital, Waterloo Road, S. E.  
 Bennett, Reginald R., B.Sc., F.I.C., Barrister-at-Law, 22-30, Graham Street, City Road, N.  
 Bennion, R., 76, St. Alban's Road, Watford.  
 Benson, Robert H., The Dispensary, Guy's Hospital, S.E.  
 Beverley, T. L., 1, Ulysses Road, West Hampstead.  
 Billington, F., 201, Edge Lane, Liverpool.  
 Bilson, F. E., 1, Lansdowne Crescent, Bournemouth.  
 Bird, F. C. J., Devon Wharf, Emmott Street, Mile End, E.  
 Black, W. J., 17, Main St., Tweedmouth, Berwick-on-Tweed.  
 Blackburn, A. E. H., o/o Messrs. Mottershead & Co., 7, Exchange Street, Manchester.  
 Blain, A. L., 69, Market Street, Manchester.  
 Blain, William Rushton, 25, Market Street, Bolton.  
 Blair, Richard, 7, Patrick Street, Cork.  
 Blake, R. F., F.I.C., F.C.S., 128, Scottish Provident Buildings, Belfast.  
 Blakely, P. L., 11, Cross Street, Ryde, Isle of Wight.  
 Blenkiron, J., 115, Princes Street, Edinburgh.  
 Blyth, Miss, Royal Infirmary, Sunderland.  
 Blyton, J. H., 76, Gordon St., Lower Broughton, Manchester.  
 Boa, Peter, 64, Morningside Drive, Edinburgh.  
 Boehm, F., 16 Jewry St., E.C.  
 Bodsworth, H., Bretton, Chester.  
 Bolton, C. A., 40, Carlton Street, Nottingham.  
 Bolton, Miss M., 36, Dover Street, Hull.  
 Bonner, Alex. C. (Messrs. W. Davidson, Ltd.), Palmerston Road, Aberdeen.

- Bonner, C. G., 20, New St., Dorset Square, N.W.  
 Boorne, H. E., 49, Woodstock Road, Redland Green, Bristol.  
 Bourdas, I., Dunoon House, 3, Nightingale Lane, Clapham Common, S.W.  
 Bourdas, Isaiah, junr., 6, Pont Street, Belgrave Square, S.W.  
 Bourne, H. Frederick, 11, Strand, Torquay.  
 Bowe, John L., 39, Cambridge St., Rugby.  
 Bowie, G. Duncan, 46, Tufnell Park Road, N.  
 Boyack, W., c/o Squire & Sons, Oxford Street, W.  
 Braithwaite, J. O., "Holme-Lacey," Warren Road, Chingford, Essex.  
 Braithwaite, Miss D. M., "Holme-Lacey," Warren Rd., Chingford, Essex.  
 Brammell R. T., 12, Ryde Vale Rd., Balham, S.W.  
 Brander, Bruce McD., 42, Cannon Street, London, E.C.  
 Branson, F. H., 14, Commercial Street, Leeds.  
 Branson, F. W., F.I.C., F.C.S., Wyncholme, Far Headingley, Leeds.  
 Breadner, C. G., 268, Waterloo Road, Cheetham, Manchester.  
 Breakspear, A. E., 6, Drayton Bridge Road, Hanwell, W.  
 Bromridge, R., 17, Bloomsbury Square, London, W.C.  
 Brewis, E. T., F.I.C., 31, Belgrave Road, Leyton, Essex.  
 Bright, R., 48, Bridge Street, Peterborough.  
 Brinson, G., 58, Greenbank Rd., Devonshire Park, Birkenhead.  
 Brinson, William C., The Laurels, Chesterfield.  
 Britton, A. B., 7-11 Aldersgate Street, E.C.  
 Brittain, E. H., 469, Holloway Road, London, N.  
 Brooks, C., 4, Northumberland Avenue, W.C.  
 Brooks, J., 42, Shudehill, Manchester.  
 Brown, Alex., 34, Leinster Terrace, W.  
 Brown, John, Exchange Buildings, Melrose.  
 Brown, Hugh, c/o Messrs. J. F. Macfarlane, 93, Abbeyhill, Edinburgh.  
 Brown, John, 3, High Street, Berwick-on-Tweed.  
 Brown, W. R., Glenash, Giffnock, Glasgow.  
 Brown, D. Rainy, Abbey Hill Chemical Works, Edinburgh.  
 Brown, David, F.R.S.E., Willowbrae House, Piershill, Edinburgh.  
 Brown, George, 14, Bingham Road, Croydon.  
 Brown, J., "Glencoe," 20, Tower Road, Dartford, Kent.  
 Browne, F., F.I.C., 64, Victoria Street, Bury St. Edmunds.  
 Browne, Wm., 509, Finchley Road, N.W.  
 Bruce, A. L., 9, Millburn Street, Ferryhill, Aberdeen.  
 Bruce, Miss G., Warneford Hospital, Leamington.  
 Brumwell, C. W., 72, Euston Square, N.W.  
 Brunker, J. E., M.A., F.C.S., 18, Grosvenor Place, Rathmines, Dublin.  
 Bryant, E. G., Northern College of Pharmacy, Burlington Street, Manchester.  
 Buchanan, D., Kirriemuir, N.B.  
 Buchanan, Margaret E., Gordon Hall, Gordon Square, W.C.  
 Buck, Anthony, S., 179, Bedford Street, Liverpool.  
 Buckingham, H., Park Road, Aston Manor, Birmingham.  
 Buckle, J., 20, Market Place, Malton, Yorks.  
 Bullen, F. E., Pharmacist, H.M. Prison, Princetown, Devon.  
 Burgess, A. H., 37, Stamford New Road, Altrincham.  
 Burr, Percy W., Oak Alyn, Gwernymynydd, Mold.

- Burrell, Thos., 48, High Street, Montrose.  
Burroughs, G. H., 58, Hanover Street, Liverpool.  
Burton, Harry, 507, Bearwood Road, Birmingham.  
Bush, Alfred W., Ash Grove Works, Hackney, N.E.  
Butler, E. H., New Haymarket, Leicester.  
Butterfield, A. E., 104, Golden Lane, E.C.
- Campkin, Alderman A. Sidney, J.P., Ty Castan, Brooklands Avenue, Cambridge.  
Carmichael, Matthew, 1103, Pollokshaws Road, Crossmyloof, Glasgow.  
Carr, C. F., 21, Long Row, Nottingham.  
Carter, J. C., 86, St. James' St., Holloway, N.  
Cave, J. R., 52, Nevill Street, Southport.  
Chamberlain, P. G., M.A., F.C.S., 3, Market Place, Rugby.  
Chapman, A. Chaston, F.I.C., 8, Duke Street, Aldgate, E.C.  
Chapman, Robert S., M.P.S.I., F.S.M.C., Medical Hall, Donegal.  
Chase, T., Five Ways, Edgbaston, Birmingham.  
Chaston, A. E., 45, High Street, Winchester.  
Chater, A. J., 17, Bloomsbury Square, W.C.  
Cheers, R. A., St. George's Hospital, S.W.  
Cheotham, P., 27, Trinity Rd., Tulse Hill, S.E.  
Cheney, Henry R., 21, High Street, Leominster.  
Chesterfield, T. M., 190, Canterbury Street, Gillingham, Kent.  
Cholerton, Alf. F., F.C.S., Wynnstead, Knighton Grange Road, Leicester.  
Christie, R. A., 414, Clarkston Road, Muirend, Glasgow.  
Church, E. H., 18, St. Andrew's Street, Cambridge.  
Clague, Thos. Maltby, A.I.E.E., 11, Grey Street, Newcastle-on-Tyne.  
Clare, Jno., 1, Harcourt Place, Scarborough.  
Claremont, Miss H. E., 81, Camden Road, N.W.  
Clark, A. J., 94, Morningside Road, Edinburgh.  
Clarke, Dr., Guy's Hospital, S.E.  
Clark, W. Inglis, D.Sc., 104, 106 & 108, South Canongate, Edinburgh.  
Clarke, F., 41, New Commercial Street, Tredegar.  
Clarke, R. Feaver, J.P., "Dancholme," Pelham Road, Gravesend.  
Clarke, W. J., Helenslea, Stanhope Rd., Stockton-on-Tees.  
Clayton, Alderman C., 158, Cowley Road, Oxford.  
Clayton, F. C., 18, St. James' Road, Birmingham.  
Cleave, J. W., 121, High Street, Notting Hill Gate, W.  
Cleworth, John, 56, Ducie Street, Oxford Road, Manchester.  
Clubb, W. H., 238, Smithdown Road, Liverpool.  
Coats, J. T., 105-107, Broughton Street, Edinburgh.  
Cofman, Joseph, 6, Bloomsbury Street, W.C.  
Cole, G. H., Blundell Sands, Liverpool.  
Coleman, J. H., 7, Worcester Street, Wolverhampton.  
Collins, H. G., Staines Road, Sunbury, Middlesex.  
Connolly, Thomas Richard, 40, Clanbrassil St., Dundalk.  
Connor, J. E., J.P., Ph.C., M.C.P.S.I., 79, Hill Street Newry.  
Cooke, C. W., Pharmacist, Hoylake.

- Cook, H. F., J.P., 83, Victoria Road, New Chesterton, Cambridge.  
Cooper, F., 4, Millman Terrace, Hornsey, N.  
Cooling, F. C., 29, Christchurch Road, Oxtou, Birkenhead.  
Cooper, G. H., 582, Oldham Road, Failsworth, Manchester.  
Cooper, L., 14, Henrietta St., Covent Garden, W.C.  
Cope, John A., 3, Market Place, Derby.  
Corfe, A. F., 5, Gabriel's Hill, Maidstone.  
Corfield, Ed., 26, Bennetts Hill, Birmingham.  
Cortis, A. B., F.C.S., 30, South Street, Worthing.  
Cornwell, T. C., Piccadilly, Hanley, Staffs.  
Coull, Dr. Geo. 5, West Claremont Street, Edinburgh.  
Cowie, William Beaverly, Principal, Edinburgh Central School of Pharmacy, 26, Clyde Street, Edinburgh.  
Craine, J. P., Northgate Pharmacy, Chester.  
Crane, J. S., Brompton Hospital, S.W.  
Crawford, D., c/o Messrs. Savory & Moore, 143, New Bond St., W.  
Cripps, R. A., F.I.C., The Laboratory, d'Avigdor Road, Hove, Sussex.  
Crombie, James, 323, Paisley Road West, Glasgow.  
Crompton, Henry, 16, Park Hills Road, Bury.  
Cross, E. R., 12, Filey Road, Scarborough.  
Cross, W. Gowen, J.P., 70, Mardol, Shrewsbury.  
Crossley-Holland, F. W., F.C.S., Menley House, Farrington Rd., E.C.  
Crossley, Prof. A. W., D.Sc., Ph.D., F.R.S., King's College, Strand, W.C.  
Crowden, S. G., 120, Marchmont Road, Edinburgh.  
Cruikshank, G. M., 19, Main Street, Turriff, Aberdeenshire.  
Cuff, J. Harcombe, 23, Wear Bay Crescent, Folkestone.  
Cumming, J., 6, Victoria Street, Crewe.  
Cummings, Wm., 49, Reform Street, Dundee.  
Currie, W. L., 223, Byres Road, Glasgow.  
Cussons, J. W., J.P., 33, High East Street, Dorchester.  
Cuthbert, R., 12, Westgate, Huddersfield.  
Cuxson, J. (Cuxson, Gerrard & Co.), Oldbury, Birmingham.
- Dall, John, 27, Bruntsfield Place, Edinburgh.  
Dalmas, W. E. de St., c/o Dalmas & Co., Leicester.  
Darling, W. H., F.I.C., F.C.S., 26, Dover Street, Oxford Road, Manchester.  
Davenport, H., 117, Union Street, S.E.  
Davies, Frank, 512, Huddersfield Road, Oldham.  
Davidson, A., 172, High Street, Montrose, N.B.  
Davidson A. Linton, 172, High Street, Montrose, N.B.  
Davidson, P., 342, High Road, Brondesbury, N.W.  
Davis, C., Leamington Spa.  
Deakin, J. W., J.P., Braeside, Northwich.  
Deane, Harold, B.Sc., F.I.C., c/o Stafford Allen & Sons, Long Melford, Suffolk.  
Deck, A. A., 9, King's Parade, Cambridge.  
Demuth, R., 68, Salisbury Rd., N.W.  
Dey, Alex. J., Blandfield Chemical Works, Wheatfield Road, Edinburgh.  
Dickie, J. G., 1, Grosvenor Terrace, Aberdeen.  
Dickinson, D., Hoole Pharmacy, Chester.

Dickson, D., 716, Ashton New Road, Clayton, Manchester.  
 Dickson, J. Scott, 48, Osborne Avenue, Newcastle-on-Tyne.  
 Dixon, P. T. (Messrs. Tozer Kemsley & Co.), 84, Fenchurch St., E.C.  
 Dixon, Prof. W. E., M.D., M.A., Pharmacological Laboratory, Cambridge.  
 Dobbin, Leonard, Ph.D., Chemistry Department, The University, Edinburgh.  
 Dobinson, T., 125, Newgate Street, Bishop Auckland.  
 Dodd, W. Ralph, F.C.S., Burton Grange, Cheshunt, Herts.  
 Dolbear, John, 108, High Street, Oxford.  
 Donaldson, G., 122, Queen Street, Portsea, Portsmouth.  
 Dott, D. B., F.R.S.E., F.I.C., Ravenslea, Musselburgh.  
 Douglas, J. Wellesley, 19, Kennington Terrace, Kennington Park, S.E.  
 Druce, G. Claridge, J.P., M.A., F.L.S., Yardley Lodge, 9, Crick Road, Oxford.  
 Duncan, W., F.C.S., Royal Dispensary, 21, West Richmond Street, Edinburgh.  
 Dunlop, Dr. Chas. J. B., M.C.P.S.I., etc., 38, Harrington Street, Dublin.  
 Dunn, C. A., 58, Bunhill Row, E.C.  
 Dunn, W. R., Oakengates, Wellington, Salop.  
 Dunstan, S., Royal Victoria Infirmary, Newcastle-on-Tyne.  
 Durrans, Thos. H., B.Sc., F.I.C., 10, Titchfield Terrace, Regent's Park, N.W.  
 Dutton, Hugh O., 2, King Street, Rock Ferry, Birkenhead.

Eacott, R. G., Green Street Sittingbourne.  
 Eardley, J. F., 265, Glossop Road, Sheffield.  
 Edwards, J. M., 92, Jamaica Road, S.E.  
 Egan, J. H., 3, Fleet Street, Liverpool.  
 Elder, A., Woodchurch Lane, Prenton, Birkenhead.  
 Elliot, W. M., High St., Coldstream, N.B.  
 Ellis, R., 3, New Wellington Terrace, Limerick.  
 Ellithorne, A. H., 26, Wellington Road, Oxtou, Birkenhead.  
 Elmitt, William, 193, Osmaston Road, Derby.  
 Elsdon, G. D., B.Sc., F.I.C., Municipal Laboratory, Regent Road, Salford.  
 Etheridge, Miss A., Beckett Hospital, Barnsley.  
 Evans, Sir Edward, J.P., 56, Hanover Street, Liverpool.  
 Evans, D. A., 6, Milsom Street, Bath.  
 Evans, D. G., 52, Sutherland Avenue, Maida Vale, W.  
 Evans, D. H., 148, Lodge Lane, Liverpool.  
 Evans, J., 116, Fitzroy Street, Cambridge.  
 Evans, J. E., 69, Leytonstone Rd., Stratford, E.  
 Evans, J. H., Medical Hall, Market Cross, Lymm.  
 Evans, J. H. E., 56, Hanover Street, Liverpool.  
 Evans, J. J., J.P., 56, Hanover Street, Liverpool.  
 Evans, J. N., 56, Hanover Street, Liverpool.  
 Evans, John, F.I.C., F.C.S., City Analyst's Laboratory, Sheffield.  
 Evans, Kenneth W., 56, Hanover Street, Liverpool.  
 Evans, W. P., 56, Hanover Street, Liverpool.  
 Evelyn, J. S., c/o Idris & Co., Northumberland Street, Liverpool.



- Ewell, R. M., 37, Townwall Street, Dover.  
 Ewing, Jas. Laidlaw, J.P., LL.D., 104, Holyrood Road, Edinburgh.  
 Exley, J., 34, Hunslet Lane, Leeds.
- Fairburn, H., Market Place, Northallerton.  
 Fairclough, R. A., c/o Lennon, Ltd., Lafone Street, S.E.  
 Fairley, T., F.R.S.E., F.I.C., F.C.S., 17, East Parade, Leeds.  
 Fairweather, J. Y., 313, Sydenham Road, S.E.  
 Fairweather, E. B., F.C.S., King's College Hospital, S.E.  
 Farr, E. H., F.C.S., The Laboratory, Uckfield, Sussex.  
 Farries, Thos., F.I.C., F.C.S., 16, Coleman Street, E.C.  
 Ferrall, A. T., 67, Lower Mount Street, Dublin.  
 Ferrier, J., 22, Lumley Street, Grangemouth.  
 Fielding, M. A., 2, Farleigh Place, Cork.  
 Fielding, P. J. D., F.C.S., F.S.M.C., 66, Patrick Street, Cork.  
 Finnemore, H., B.Sc., F.I.C., Guy's Hospital, S.E.  
 Flanders, Henry, 104, Mill Road, Cambridge.  
 Fletcher, F. W., F.C.S., c/o Fletcher, Fletcher & Co., Ltd., Vibrona Laboratories, Holloway, N.  
 Fletcher, H. G., Dyserth, N. Wales.  
 Flick, W. S., 48-50, Southwark Street, S.E.  
 Foggan, George, Leadgate House, Bedlington, Northumberland.  
 Forbes, John J., F.S.M.C., 7, Scott Street, Perth.  
 Ford, Miss, Pharmacist, Kirriemuir.  
 Forret, J. A., 26, Brougham Place, Edinburgh.  
 Forrester, A., 312, Cathcart Road, Glasgow.  
 Forrester, James, M.B., C.M., 312, Cathcart Road, Glasgow.  
 Foster, John, 479, Sauchiehall Street, Glasgow.  
 Foster, Murray Toogood, Collumpton, Devon.  
 Foster, Reginald Le Neve, J.P., F.C.S., Fulshaw Cottage, Wilmslow, Cheshire.  
 Fouracre, R., 73, Welbeck Street, W.C.  
 Fox, C. E., J.P., 109-113, Bethnal Green Road, E.  
 Fox, T. B., 50, Wigmore St., W.  
 France, J. H., 47, Ouseley Rd., Wandsworth Common, S.W.  
 Francis, Alan, 22-30, Graham Street, City Road, N.  
 Francis, Geo. Bult, 22-30, Graham Street, City Road, N.  
 Francis, James B., 53, Hope Street, Wrexham.  
 Francis, Wm. Hy., 150, York Road, Lambeth, S.E.  
 Franklin, J. H., Sunnyside, Broom Lane, Higher Broughton, Manchester.  
 Fraser, A., 99, High Street, Forres.  
 Fraser, Alexr., 100, High Street, Paisley, N.B.  
 Fraser, G., 4, London Street, Reading.  
 Froeman, Thos., 21, Goldswong Terrace, Nottingham.  
 Frost, J. H., 9, High St., Hornsey, N.  
 Fudge, C. W., Shepton Mallet.  
 Furnival, S., 265, Glossop Road, Sheffield.
- Gadd, H. Wippell, Barrister-at-Law, 100, Fore Street Exeter.  
 Galloway, P. H., 154-162, Walworth Road, S.E.  
 Gamble, F. W., 7, Vere Street, W.  
 Gange, G., Claremont, Hermon Hill, Snaresbrook, Essex.  
 Gardner, Miss L. B., East Dispensary, Liverpool.  
 Garnett, Henry, F.C.S., 32, Dover Street, Oxford Road, Manchester.

- Garrow, W., Allerton Newhouse, Stirling  
 Garsed, Wm., F.I.C., 15, Gascoyne Road, Hackney, N.E.  
 Gaze, W. E., 10, The Avenue, Highams Park, N.E.  
 Gerrard, A. W., F.C.S., Southmead, St. Agnes Road, Moseley  
 Gibson, F. J., 93, Darlington Street, Wolverhampton.  
 Gibson, G. W., St. Pancras South Infirmary, Pancras Road, N.W.  
 Gibson, R., Erskine Street, Hulme, Manchester.  
 Gibson, W. H., F.C.S., 122, King's Road, Brighton.  
 Gifford, R. L., Blackburn.  
 Gilderdale, F., F.C.S., c/o John Ismay & Sons, Newcastle on Tyne.  
 Giles, W., J.P., 123, Crown Street, Aberdeen.  
 Gill, Jos. W., 567, Liverpool Street, Pendleton, Manchester.  
 Gilling, C., F.I.C., Bagshot Road, Sunningdale.  
 Gilmour, J. P., 312, Cathcart Road, Glasgow.  
 Gittoes, Samuel J., 56, Lower High Street, Wednesbury.  
 Glass, W. S., 193, Morningside Road, Edinburgh.  
 Gledhill, R., 61, Westgate, Dewsbury.  
 Glew, F. H., 156, Clapham Road, London S.W.  
 Glyn-Jones, Alderman W. S., Barrister-at-Law, Endsleigh, Palmer's Green, London, N.  
 Goldby, F., The Enfield Pharmacy, Enfield Town, N.  
 Goldon, H. V., Chemist, B-rr, King's County, Ireland.  
 Goldthorpe, Arthur, 70, Herbert Road, Plumstead, S.E.  
 Goodall, F. C., 72, Great Russell St., W.C.  
 Goodall, T. S., 33, Essendine Road, Maida Vale, W.  
 Goodyer, N. S., 20, Old Buildings, Lincoln's Inn, W.C.  
 Goss, J. O., Royal Berks Hospital, Reading.  
 Gough, J. H., F.C.S., 4, Woodland Grove, Newton Road, Leeds.  
 Grant, John, Methlick, Aberdeenshire.  
 Grassick, Alex., 82, High St., Hornsey, N.  
 Green, J., 262, New Cross Road, S.E.  
 Greenish, Prof. H. G., F.I.C., 17, Bloomsbury Square, W.C.  
 Groy, J. E., 39, Marchmont Crescent, Edinburgh.  
 Grieb, Christopher M. W., B.Sc., F.I.C., Woodpark Cottage, Polmont, Stirlingshire.  
 Grier, Jas., M.Sc., Pharmaceutical Dept., The University, Manchester.  
 Griffiths, R. E., B.Sc. A.I.C., 20, Baldwyn Gardens, Acton, W.  
 Griffiths, W., 134, Market Place, Cirencester.  
 Grimes, Henry C., Maryville, Carysfort Avenue, Blackrock, Co Dublin.  
 Grimwade, E. W., St. John's House, 124-127, Minories, London, E.  
 Groves, R. H., "Westfield," Park Road, Blandford.  
 Gulliver, W. F., 6, Lower Belgrave Street, Pimlico, S.W.  
 Guthrie, Thos., 204, Glasgow Rd., Clydebank, N.B.  
 Gwatkin, J. R., 49, Grand Parade, Brighton.
- Haigh, A., Royal Infirmary, Halifax.  
 Hall, John W., 6, Long Causeway, Peterboro'.  
 Hall, S. Godfrey, F.C.S., c/o Messrs. Edward Cook & Co., Ltd., Bow, E.  
 Haller, Geo., 52, Leadenhall Street, E.C.  
 Hammond, H. W., The Dispensary, St. Bartholomew's Hospital, E.C.  
 Hammett, T., 38, South Road, Liverpool.

- Hampshire, C. H., B.Sc., F.I.C., University College Hospital,  
Gower Street, W.C.
- Hanbury, F. Capel, The Gables, Forty Hill, Enfield, N.
- Hanbury, F. J., F.L.S., 37, Lombard Street, E.C.
- Hancock, S. R., 96, Howard Road, Leicester.
- Hankinson, G. R., High Street, Uttoxeter.
- Hanson, Arthur, 3, High Street, Queensbury, Bradford, Yorks.
- Happolt, C., 182, Westcombe Hill, Blackheath, S.E.
- Harbin, G. A., 23, Whitewell Road, Southsea.
- Hardwick, Stewart, 21, Commercial Road, Bournemouth.
- Harkness, John, Custom House Drug Stores, Victoria Docks, E.
- Harkness, John, 20, Minto Street, Edinburgh.
- Harley, T., 21, High Street, Perth.
- Harmer, G. A., 47-49, South Street, Eastbourne.
- Harper, T., 39, Camden St., Belfast.
- Harrie, H. W., Devon Wharf, Mile End, E.
- Harrington, J. F., 15, Kensington High Street, W.
- Harrison, E. F., B.Sc., F.I.C., 57, Chancery Lane, W.C.
- Harrison, Ald. J., 33, Bridge Street, Sunderland.
- Harrison, R. Casswell, The Pharmacy, Grayshott, Surrey.
- Harrison, W. B., 6, Bridge Street, Sunderland.
- Harvard, H. L., 95, Bryn-y-Mor Road, Swansea.
- Harvey, F., 1, Claremont Rd., Surbiton.
- Harvey, H. M., F.I.C., 151, Coventry Rd., Ilford, Essex.
- Harvey, J. W., 31, Gt. George's St., Cork.
- Harvey, S., F.I.C., F.C.S., South Eastern Laboratory, Canterbury.
- Hawley, J. W., 216, Aigburth Road, Liverpool.
- Hay, W. F., 476, Union Street, Aberdeen.
- Haycock, J., F.C.S., Hill Top House, Great Glen, near Leicester.
- Hayhoe, W., 45, Cromwell Road, Pokesdown, Bournemouth.
- Heap, J. H., 34, Cheapside, Hanley.
- Hearle, J., 33, Liverpool Rd., N.
- Heddle, J. S. B., 257A, Leith Walk, Edinburgh.
- Henderson, H. J., 1, Payne's Park, Hitchin.
- Hendry, R. L., 27, Earl Grey Street, Edinburgh.
- Hennings, C. R., Ph.D., 19, St. Dunstan's Hill, E.C.
- Henry, James, 24, Bank Street, Galashiels.
- Herd, H., St. Mary's Place, Newcastle-on-Tyne.
- Heslton, C. J., Brooklands, Gosforth, Newcastle-on-Tyne.
- Hewitt, H., The Dispensary, 53, Clerkenwell Rd., E.C.
- Howitt, Silas, The Avenue Pharmacy, Ashton-under-Lyno.
- Hewitt, T. L., 136, Queen Victoria Street, E.C.
- Hewlett, John C., F.C.S., 35-42, Charlotte Street, Great Eastern Street, E.C.
- Hewlett, V. C., 40, Charlotte Street, Great Eastern Street, E.C.
- Heywood, Miss S. J., Gordon Hall, Gordon Square, W.C.
- Hicks, W. T., 28, Duke Street, Cardiff.
- Higgs, A., J.P., Richmond Rd., Kingston-on-Thames.
- Hill, C. A., B.Sc., F.I.C., 22-30, Graham Street, City Road, N.
- Hill, E., 53, Carlisle Terrace, Bradford.
- Hill, J. Rutherford, 36, York Place, Edinburgh.
- Hill, John S., 1, Academy St., Warrington.
- Hill, J. Stableford, 55, Northumberland Street, Newcastle-on-Tyne.
- Hills, J. Stuart, F.I.C., F.C.S., Oxford Works, Tower Bridge Road, S.E.
- Hills, Walter, F.C.S., 50, Wigmore Street, W.

- Hinks, Edward, B.Sc., F.I.C., 16, Southwark Street, S.E.  
 Hinkman, J., 17, High Street, Carlisle.  
 Hipwell, S. J., 67, Minster Rd., Cricklewood, N.W.  
 Hirst, Benj., Millgarth Mills, Leeds.  
 Hobbs, A. E., 33, Mount Pleasant, Tunbridge Wells.  
 Hocking, F. A., B.Sc., London Hospital, E.  
 Hodgkinson, Charles, 22-30, Graham Street, City Road, N.  
 Hogg, J. Fawcett, 102, Bedford Street, North Shields.  
 Hogg, S., 110, Shankill Road, Belfast.  
 Holding, J., 152, Hemingford Rd., Barnsbury, N.  
 Holford, S. S., 86, Allerton Road, Liverpool.  
 Hollick, R., 268, Burbury St., Lozells, Birmingham.  
 Holmes, E. M., F.L.S., Ruthven, Sevenoaks.  
 Hooper, David, L.L.D., F.I.C., F.C.S., 14, Victoria Park, Weston-super-Mare.  
 Hope, J., 332, Deansgate, Manchester.  
 Hopkinson, W. J., 82, Southwark Bridge Road, S.E.  
 Hopley, John H., 6, Northgate Street, Chester.  
 Hora, T. T., 346, York Rd., Wandsworth, S.W.  
 Hornby, F. W., 132A, Christchurch Road, Boscombe, Bournemouth.  
 Horniblow, Miss K. N., 4, Queen's Buildings, Llandudno.  
 Horsfield, F., Swanland House, Swanland Avenue, Bridlington.  
 Hoseason, J. H., Sun Buildings, Bridge Street, Manchester.  
 Hough, R., 2, Bridge Street, Manchester.  
 Howard, D., F.I.C., F.C.S., J.P., Devon House, Buckhurst Hill, Essex.  
 Howard, D. Lloyd, F.C.S., Uphall Works, Ilford, E.  
 Howell, A., Hackney Union Dispensary, Dalston, N.E.  
 Howie, W. L., F.R.S.E., 22-30, Graham Street, City Road, N.  
 Hughes, D., Chemist, Mold.  
 Hughes, W. Griffiths, 83, Blackfriars Road, Salford, Manchester.  
 Humphrey, John, 17, Bloomsbury Square, W.C.  
 Humphreys, G., High Street, Northwich.  
 Hunt, F. Wm., 106, Old Town Street, Plymouth.  
 Hunt, H. J., 65, High Street, Dundee.  
 Hunter, Andrew, 139, Princess Street, Edinburgh.  
 Hunter, Robert, 118, Union Street, Aberdeen.  
 Huskisson, H. O., F.I.C., F.C.S., F.L.S., Moon Street, Islington, London, N.  
 Hutcheon, J., 28, Charles Street (City), Manchester.  
 Hutton, John, 8, High Street, Brechin.  
 Huxtable, Chas., 34, Hanover Street, Liverpool.  
 Idris, T. H. Williams, F.C.S., 120, Pratt St., Camden Town, N.W.  
 Idris, W. T., 120, Pratt Street, Camden Town, N.W.  
 Inman, G., 44, Dale Street, Liverpool.  
 Inman, W., 11, Newbattle Terrace, Edinburgh.  
 Inman, W. C., 11, Newbattle Terrace, Edinburgh.  
 Innes, David, 47, Melbourne Street, Stalybridge.  
 Irvine, Peter, 114, Old Hall St., Liverpool.  
 Ismay, Reginald, City Road, Newcastle-on-Tyne.

Jacks, D. R., 161, Gower Street, W.C.  
 Jackson, H., 13, South Charlotte Street, Edinburgh.  
 Jackson, Prof. H., F.I.C., 49, Lansdowne Road, W.  
 Jackson, J. C., School Lane, Liverpool.  
 Jackson, J. G., 338, Abbeydale Road, Sheffield.  
 Jackson, R. E., York Villa, Shepherds Lane, Dartford.  
 James, Clarence Hilyer, 5, Promenade, Cheltenham.  
 James, J., 18, Wilton Road, Victoria, S.W.  
 Jamieson, Martin, 6, Gilmore Place, Edinburgh.  
 Jamison, W., M.C.P.S.I., Town Hall Street, Belfast.  
 Jenkins, A. H., 54, Wray Crescent, Tollington Park, N.  
 Jennings, Cornelius, Coventry Road, South Yardley, Birmingham.  
 Jennings, J. A., St. Thomas' Hospital, S.E.  
 Jensen, Harold Rupert, 6, Belvidere Road, Wallasey.  
 Johnson, R. C., Old Market Place, Grimsby.  
 Johnston, Dr. W. Vincent, 3, Cullenswood Terrace, Ranelagh, Dublin.  
 Johnstone, C. A., c/o Messrs. Woolley, Sons & Co., Victoria Bridge, Manchester.  
 Johnstone, Walter, Cromarty, N.B.  
 Jones, A., 78, Long Acre, W.C.  
 Jones, Clenyg, Glyn, Shavington Avenue, Chester.  
 Jones, E., Kilburn Lane, West Kilburn, N.W.  
 Jones, E. Oswald, 74, King Street, Brynmawr, Breconshire.  
 Jones, Edmund, Miles Bank, Hanley.  
 Jones, E. W. T., F.I.C., F.C.S., Public Analyst, 10, Victoria Street, Wolverhampton.  
 Jones, H. Humphreys, Colquitt Street, Liverpool.  
 Jones, John, 301, Edge Lane, Liverpool, and c/o W. & J. Fergusson, Strand Street, Liverpool.  
 Jones, J. P., 629, Smithdown Road, Liverpool.  
 Jones, M. I., 4, Thayer St., Manchester Sq., W.  
 Jones, W. A., West India House, Liverpool.  
 Jowett, H. A. D., D.Sc., 5, Miskin Road, Dartford, Kent.  
 Joyce, T. G., B.Sc. (Lond.), F.I.C., F.C.S., Lyttleton Works, Bromford Lane, West Bromwich.  
 Judd, W., 150, Holborn, W.C.

Kay, J. P., 205, Union Street, Aberdeen.  
 Kay, Samuel, St. Petersgate, Stockport.  
 Keall, J., 68, West Hill, Wandsworth.  
 Keith, A. R., 6, Crisp St., Poplar, E.  
 Kelly, A. E., 284, Causewayside, Edinburgh.  
 Kelly, G. J., 308, Stanley Road, Bootle.  
 Kemp, H., Chorlton-cum-Hardy, Manchester.  
 Kennedy, G., 2, Park Road, Liverpool.  
 Kennett, John Nash, Uplands, Weybridge.  
 Kerfoot, E. H., Springwood Hall, Bardsley, Lancs.  
 Kerfoot, T., J.P., Pole Bank Hall, Gee Cross, Cheshire.  
 Kerse, Wm., c/o John Ismay & Sons, City Road, Newcastle-on-Tyne.  
 Kidd, J. C., 551, Cheetham Hill Road, Manchester.  
 Kiloh, James, 108, Patrick Street, Cork.  
 Kinch, Ed., F.I.C., F.C.S., Prof. of Chemistry, Royal Agricultural College, Cirencester.  
 King, Miss, Stanley Hospital, Liverpool.

Kingzett, C. T., F.I.C., F.C.S., The Sanitas Co., Ltd., Looksley St., Limehouse, E.  
 Kirkby, W., M.Sc., F.C.S., F.L.S., Winster House, Thornfield Rd., Heaton Moor, Stockport.  
 Kluge, H. J., 13, Curzon Street, W.  
 Knight, G. J., 452, Edgware Road, W.  
 Knights, J. West, F.I.C., F.C.S., County Laboratory, 67, Tenison Road, Cambridge.  
 Knott, Herbert, 462, Blackburn Road, Bolton.  
 Knott, P., 1, Blackburn Road, Bolton.

Lacey, W. J., St. Giles Street, Norwich.  
 Lake, J. Hinton, 41, High Street, Exeter.  
 Lancaster, J., Leeds Public Dispensary, Leeds.  
 Lane, W., 8, Albert Road, Whalley Range, Manchester.  
 Last, G. V. C., 157A, Lodge Lane, Liverpool.  
 Latchmore, A., Chiltern Road, Hitchin.  
 Latreille, A., 48, Baker Street, Portman Square, W.  
 Lawrence, H., Kenley, Surrey.  
 Lawson, A. E., 60, Exmouth St., W.C.  
 Lawson, W., Bellshill, Lanarkshire.  
 Laycock, W. F., 157, Earle Road, Liverpool.  
 Layman, E. B., 50, Southwark St., S.E.  
 Layman, F. N., 48 & 50, Southwark Street, S.E.  
 Lee, S. Wright, 6, 8, & 10, Whitechapel, Liverpool.  
 Lemmon, G. U., 16, Rodney Street, Edinburgh.  
 Lennox, James, 6, Queen Margaret Place, North Kelvinside, Glasgow.  
 Lenton, Walter H., Hunstanton.  
 Lescher, F. Harwood, F.C.S., 8, Prince Edward Mansions, Palace Court, W.  
 Lescher, T. E., 60, Bartholomew Close, E.C.  
 Leslie, Robt., 627, George Street, Aberdeen.  
 Lester, J. H., 5, Grange Drive, Monton Green, Eccles.  
 Lester, T. R., 107, Patrick Street, Cork.  
 Levis, John Edwin, The Medical Hall, Skibbereen, Co. Cork.  
 Lewis, S. Judd, Ph.D., B.Sc. (Lond.), F.I.C., The Laboratories, Staple Inn Buildings, High Holborn, W.C.  
 Lincolne, W., High Street, Ely.  
 Lindley, L. S., 10, Chester Place, Hyde Park Square, W.  
 Litherland, W. H., Wallgate, Wigan.  
 Liverseegee, J. F., F.I.C., 44, Broad Street, Birmingham.  
 Livesley, T. H., Station Road, Hayfield.  
 Lloyd, H., 260, Upper St., Islington, N.  
 Lloyd, B. C., Marsh Lane, Bootle.  
 Lloyd, I. T., 267, King's Rd., Chelsea, S.W.  
 Lloyd, T. M., 101, Holt Road, Liverpool.  
 Lookyer, Conrad W., M.R.C.S., L.R.C.P., Bridgwater Lodge, Epple Bay Road, Birchington-on-Sea, Kent.  
 Lothian, John, 2, Argyll Street, Portobello, N.B.  
 Low, J. H., Broad Street, Fraserburgh.  
 Lowe, H., 18, Hough Green, Chester.  
 Lownsborough, R. E., 35, Clifton Rd., Maida Vale, W.  
 Lowther, Tom W., 131, Alcester Road, Moseley, Birmingham.  
 Loxley, F. L. K., 66, St. Giles' Street, Oxford.

Lucas, E. W., F.I.C., F.C.S., Oxford Works, Tower Bridge Road, S.E.

Lucas, H., F.C.S., 186, Clapham Road, S.W.

Lunan, George, F.C.S., 20, Queensferry Street, Edinburgh.

Maben, T., F.C.S., 19, Great Pulteney Street, W.

Macdonald, A., 9-11, Moor Lane, Fore Street, E.C.

MacEwan, P., F.C.S., 64, Southwood Lane, Highgate N.

Macfarlane, M., 19, East High Street, Forfar.

Macintyre, John, 34, High Street, North Berwick.

Mackay, G. D., Canning Street, Edinburgh.

Mackay, G., c/o Messrs. Hatrick & Co., Wholesale Chemists, Glasgow.

Mackenzie, Donald, 22, City Road, E.C.

MacLennan, Roderick, 84, Corniston Road, Edinburgh.

MacSweeney, Eugene, Ph.C., F.I.O., 91, Patrick Street, Cork.

McAdam, R., J.P., 32, Virginia Street, Glasgow.

McClelland, J., Medical Hall, Ballyshannon.

McDonald, W. G., 31, High Street, Inverness.

McDiarmid, Fraser, 4, Mayville Gardens, Trinity, Edinburgh.

McKenzie, J. M., 45, Forrest Rd., Edinburgh.

McMillan, Anthony, 623, New City Road, Glasgow.

McMullan, Thomas W., 42, Victoria Street, Belfast.

McMurray, P. B., 37, West Clyde St., Helensburgh.

McInroy, Jas., Sefton Park, Liverpool.

McRae, C., 24, Dove Road, Aintree, Liverpool.

McWalter, J. C., M.A., M.D., D.P.H., F.F.P.S. (Glas.), 19, North Earl Street, Dublin.

Mair, Wm., F.C.S., 37, Morningside Drive, Edinburgh.

Makepeace, A. B., 11, Kirkdale, Sydenham, S.E.

Mallett, T. J., 66, Victoria Park, Cambridge.

Malone-Barrett, F., St. Mary's Road, Ealing, W.

Mann, Ernest W., 19, Lower Priory, Birmingham.

Marchant, D., 1, Rockingham Place, Old Eastbourne.

Maries, C. A., South Road, Waterloo, Liverpool.

Marr, Wm., 34, Woolmanhill, Aberdeen.

Marris, G. W., 56, Hanover St., Liverpool.

Marsden, G., 221, Chester Road, Manchester.

Marsden, H. H., 16, County Road, Liverpool.

Marsden, Prosper H., F.C.S., The University, Liverpool.

Marsh, A. E., 33, Portland Road, Leicester.

Marshall, H. B. K., 133, Fenchurch St., E.C.

Marshall, John D., 50, Wigmore St., W.

Martin, H., 6, Berton Hill, Aylesbury.

Martin, J. B., Chemist, Helston, Cornwall.

Martin, N. H., J.P., F.R.S.E., F.L.S., F.C.S., Ravenswood, Low Fell, Gateshead-on-Tyne.

Martin, William, M.A., M.D., West Villa, Akenside Terrace, Newcastle-on-Tyne.

Martindale, W., Ph.D., 10, New Cavendish Street, W.

Maskew, W. J., Town Lodge, Clacton-on-Sea.

Masson, H., The London Road Pharmacy, Forest Hill, S.E.

Mather, J. H., J.P., Godalming.

Matthews, C. W., 6, Fortress Road, N.W.

Matthews, Harold E., 30, The Mall, Clifton, Bristol.

Matthews, H. R., 61, Charlotte St., Tottenham Court Road, W.

Matthews, J. H., Lyncroft, Bushwood, Leytonstone, N.E.

Maw, A. T., 11, Aldersgate Street, E.C.

- Maxwell, A. T., 51, Penny St., Lancaster.  
 Maxwell, John, Prior & Co., Londonderry.  
 Mayne, A., 7, Pembroke Street, Cork.  
 Melhuish, A. R., 78, Duke St., Grosvenor Sq., W.  
 Mercer, F. N., 101, Mostyn Street, Llandudno.  
 Merson, Geo. F., F.C.S., c/o Messrs. J. F. Macfarlan & Co., Abbey Hill, Edinburgh.  
 Michie, C. C., 175, Kentish Town Rd., N.W.  
 Michie, D. C., High Street, Hawick.  
 Middleton, A., 25, Lister Gate, Nottingham.  
 Millard, E. J., F.C.S., F.R.M.S., 35-42, Charlotte Street, E.C.  
 Miller, H., 389, High Street, Cheltenham.  
 Miller, W. E., 46, Clarendon Square, N.W.  
 Millhouse, Edw., 26, New Road, Gravesend.  
 Millidge, Alfred, 47, High Street, Newport, Isle of Wight.  
 Milling, J., Northgate St., Chester.  
 Mills, H. A., 3, Croxted Road, Dulwich, S.E.  
 Milne, F. L., 26, Clyde Street, Edinburgh.  
 Milner, J., 209, Trafalgar Rd., Greenwich.  
 Milsom, F. E., 35, Elvendon Rd., Palmers Green, N.  
 Mitchell, H., 179, Earham Grove, Forest Gate, S.E.  
 Mitchell, J. B., 58, South Clerk Street, Edinburgh.  
 Mitchell, W. G., 431, George Street, Aberdeen.  
 Moffatt, W. J., 741, Pollokshaws Rd., Strathbungo, Glasgow.  
 Montgomery, Johnston, 147, Royal Avenue, Belfast.  
 Moore, J. Langford, St. Bartholomew's Hospital, E.C.  
 Morgan, Henry Brunt, 5, Cavendish Rd., Blindellsands, Liverpool.  
 Morrell, F. G., Ph.D., The Sir John Cass Institute, Aldgate, London, E.C.  
 Morris, G., Union Street, Wednesbury.  
 Morson, Albert, 14, Elm Street, Gray's Inn Road, W.C.  
 Morson, L. J., 14, Elm Street, Gray's Inn Rd., W.C.  
 Morson, Thomas D., 14, Elm Street, Gray's Inn Rd., W.C.  
 Morson, T. Pierre, 14, Elm Street, Gray's Inn Road, W.C.  
 Mumford, H. G., Woodlands, Linden Road, West Green, N.  
 Mundell, S. R., 178, Harehills Avenue, Leeds.  
 Murchie, John, 12, Bridgeton Cross, Glasgow.  
 Murray, A. A. 133, Bruntsfield Place, Edinburgh.  
  
 Naylor, Frank, 11, Aldersgate St., E.C.  
 Naylor, W. A. H., F.I.C., 22-30, Graham Street, City Road, N.  
 Neale, H. H., 4, Red Cross Street, E.C.  
 Neathercoat, E. T., J.P., Church Street, Weybridge.  
 Needham, Thos. 24, Clarendon Court, Maida Vale, W.  
 Nelson, W. B., 43, Hunters Road, Harrow.  
 Nesbit, James, 236, High Street, Portobello.  
 Newsholme, G. T. W., J.P., F.C.S., 27, High St., Sheffield.  
 Nicholl, I. W., M.P.S.I., 25, High Street, Belfast.  
 Nicholson, J. H., Maxwelltown, Dumfries.  
 Nicoll, R., 8, Roseneath Street, Edinburgh.  
 Nicolson, D. W., 17, Smith's Place, Edinburgh.  
 Nightingale, J. C., Hebron, Fitzjohn Avenue, Barnet, Herts.  
 Nimmo, W., The Pharmacy, Pallion, Sunderland.  
 Noble, Harry W., 24, Newgate Street, Newcastle-on-Tyne.  
 Noble, J., 40, Essex Road, Islington, N.  
 Norwood, J. P., Wath-on-Dearne, nr. Rotherham.  
 Nuthall, E., Bank Plain, Norwich.



- Ord, S. W., 3, Hanover Street, Hanover Square, W.  
 Orrell, W. P., 9, High Street, Rushden, Northants.  
 Ottey, Thomas, King's Court, Colmore Row, Birmingham.  
 Ough, Lewis, F.L.S., F.C.S., "Fernleigh," St. James' Rd., Leicester.  
 Overton, Percy S., 50A, Lord Street, Liverpool.  
 Owen, R. Cecil, B.Sc., 89, Foregate Street, Chester.
- Pack, F. J., 38, The Avenue, Hitchin.  
 Paddock, S. E., 516, Stanley Road, Bootle.  
 Palmer, F. J., 12, Montpellier Avenue, Cheltenham.  
 Park, C. J., 23, Mutley Plain, Plymouth.  
 Parker, E., Pharmacist, Scarborough.  
 Parker, R. H., F.C.S., "Ravenscar," Orchard Rd., Blackheath, S.E.  
 Parkes, Albert E., F.I.C., F.C.S., Chemical Dept., 43, White Horse St., Stepney, E.  
 Parkes, G. J. R., Ferndale, 109, Musters Road, West Bridgford, Nottingham.  
 Parkinson, F. W., Atherstone, Warwickshire.  
 Parrott, J., 50, Friars Stile Rd., Richmond, Surrey.  
 Parry, E. J., B.Sc., F.I.C., F.C.S., 56A, Great Dover St., S.E.  
 Parry, L. Moreton, 163, Oakfield Road, Liverpool.  
 Parsons, Miss D. M., 29, London Rd., Sevenoaks.  
 Paterson, A. G. C., c/o Hopkin & Williams, Lavender Mount, Ilford.  
 Patey, W. J., c/o Roberts & Co., New Bond Street, W.  
 Paton, A. H., Sherwood Pharmacy, Glasgow Rd., Paisley.  
 Patterson, George Rae, Milburn Road, Ashington, Northumberland.  
 Pearson, G. E., 240, Goswell Road, E.C.  
 Pearson, G. E., Snow Hill Buildings, E.C.  
 Peck, E. Saville, M.A., 30, Trumpington Street, Cambridge.  
 Peck, J. Wicliffe, Hospital for Sick Children, Gt. Ormond Street, W.C.  
 Pentney, J. C., Waveney House, Cazenove Rd., Upper Clapton, N.  
 Perry, Sir Cooper, M.D., Superintendent's House, Guy's Hospital, S.E.  
 Pescod, Wm., 60, Osborne Avenue, Newcastle-on-Tyne.  
 Phillips, A. J., 156, Cromwell Road, South Kensington, S.W.  
 Phillips, H. A., A.I.C., 17, Bloomsbury Square, W.C.  
 Phillips, H. S., 17, Bloomsbury Square, W.C.  
 Phillips, J., J.P., Thornhill, Wigan.  
 Phillips, P. B., 2, High Street, Woking.  
 Phillips, Professor R. W., D.Sc., The University, Bangor.  
 Phillips, Sidney, 8, Lichfield Street, Wolverhampton.  
 Philp, W. J. I., 34, High Street, Notting Hill Gate, W.  
 Pickering, John, 20, Church Road, Stanley, Liverpool.  
 Pidd, A. J., Herrestead, Wilbraham Road, Manchester.  
 Pinchbeck, Gerald, F.C.S., 72, Gresham Road, Middlesbrough.  
 Pinchen, W. J., 20, St. James Parade, Muswell Hill, N.  
 Pinnington, A., 3, St. Edmond's Road, Bootle.  
 Pirrie, Geo., Milltown, Rothiemay, N.B.  
 Pitman, J., 42, Redcliff Hill, Bristol.  
 Platts, John, 19, Lower Priory, Birmingham.

Poble, J., 13, Great Hampton St., Birmingham.  
 Pollard, Evelyn Wm., B.Sc., 23, High Street, Ryde, I.W.  
 Poole, Weston, 47, High Street, Newcastle, Staffs.  
 Potter, Alderman H., 60/64, Artillery Lane, E.  
 Potter, W., High Street, Plaistow, E.  
 Pratt, W. R., B.Sc., A.I.C., 17, Bloomsbury Square, W.C.  
 Prebble, E., 187, Kensington, Liverpool.  
 Prebble, J. G., Chislehurst, Kent.  
 Presant, C. S., 258, Euston Road, N.W.  
 Preston, Job, 105, Barker's Pool, Sheffield.  
 Preston, Thos. I., Oakwood Pharmacy, Roundhay, Leeds.  
 Price, H. H. G., 15, Kensington High Street, W.  
 Probyn, Lt.-Col. Clifford, D.L., J.P., 55, Grosvenor Street, Grosvenor Square, W.  
 Procter, Henry Raithby, 113, The Grove, Hammersmith, W.  
 Purse, A. D., 15, Salem Street, Bishopwearmouth.

Quant, Ernest, F.C.S., 2, Park Crescent, Torquay.  
 Quarroll, Wm. Henry, M.A., 3, East India Avenue, E.C.

Radford, J. A., 14, Union Street, Birmingham.  
 Rae, J. Spence, 17, Park Place, E. Sunderland.  
 Ramage, A. J., 2, Burleigh Villa, Brighton Rd., Addlestone.  
 Ranken, C., F.C.S., F.R.M.S., 19, Stockton Road, Sunderland.  
 Rankin, W. J., 11, Waring Street, Belfast.  
 Ransom, F., F.C.S., The Chilterns, Hitchin.  
 Ratcliffe, Samuel, 659, Lord Street, Southport.  
 Reekie, B., 18, Brandon St., Hamilton, N.B.  
 Rees, R. P., M.R.P.S., Ifor Cottage, Dowlais, Glam.  
 Reid, John F., F.I.C., Hazeldene, Melksham, Wilts.  
 Reid, A., 312, Cathcart Road, Glasgow.  
 Reith, John Reid, The Square, Cults, by Aberdeen.  
 Remington, J. S., F.C.S., The Laboratory, "Aynsome," Grange-over-Sands.  
 Renouf, Miss N., 29, Stanley Gardens, W.  
 Richards, J. H., 11, Mellor Road, Prenton, Birkenhead.  
 Richards, P. A. Ellis, F.I.C., F.C.S., Royal Dental Hospital, W.C.  
 Richardson, F. W., 18, Comiston Road, Edinburgh.  
 Richardson, F. W., F.I.C., F.C.S., Thorp Chambers, Hustlergate, Bradford, Yorks.  
 Richardson, H. N. B., B.A., F.C.S., c/o Messrs. John Richardson & Co., 10, Friar Lane, Leicester.  
 Richardson, J. W., Alyn Bank, Pontblyddyn, near Mold, N. Wales.  
 Righton, J., 515, Lord Street, Southport.  
 Rivers-Wilson, A., Union Society, Oxford.  
 Roberts, R., 13, Church Street, Camberwell, S.E.  
 Roberts, W., 12, Cullam Street, E.C.  
 Roberts, W. G., 441, Smithdown Road, Liverpool.  
 Robertson, R., 55, Moodie St., Dunfermline.  
 Robertson, D. Stewart, 170, Main Street, Rutherglen, N.B.; and 70, Caledonia Road, Glasgow.  
 Robertson, John, 19, West Port, Arbroath, N.B.  
 Robertson, Dr. J. McGregor, M.A., M.B., 28, Buckingham Terrace, Great Western Road, Glasgow.

Robins, H. H., c/o Chas. Southwell & Co., Ltd., Jacob Street, Dockhead, S.E.  
Robinson, P., Oxford House, Mile End Road, Norwich.  
Robinson, Sir T. W., J.P., 112, Upper George's St., Kingstown, Dublin.  
Rodwell, Henry, St. Thomas' Hospital, S.E.  
Rogers, Frank A., 327, Oxford Street, W.  
Roper, H. C., 29, Mosley Street, Newcastle-on-Tyne.  
Ross, Andrew L., Monterey, Castle Terrace, Bervie, Scotland.  
Rowland, George Howard Charles, 7, Castle Street, Edinburgh.  
Rowland, E. Osmond, 7, Castle Street, Edinburgh.  
Rowsell, P. F., 74, High Street, Exeter.  
Royal, A., 20, Kingfield Road, Orrell Park, Liverpool.  
Royle, J., 285, Duke Street, Glasgow.  
Royle, John W., 45, Belvedere Road, S.E.  
Russell, C. J., Jesmond Rd., Newcastle-on-Tyne.

Sage, C. E., F.I.C., F.C.S., 10, London St., Fenchurch St., E.C.  
Salter, L. E., 7, The Broadway, West Norwood.  
Sambrook, John T., 59, High Street, Barnet.  
Sampson, J. W., F.C.S., Cleveland Pharmacy, Bath.  
Sanford, W., 25, Campion Rd., Putney, S.W.  
Sanger, E., 258, Euston Rd., N.W.  
Sargeant, F. Pilkington, F.C.S., College of Pharmacy, Clarendon Road, Leeds.  
Saunders, S. T., 98, St. John St., E.C.  
Saunders, W. G., 34, Hanover Street, Liverpool.  
Saunders, W. H., 34, Hanover Street, Liverpool.  
Savage, F. C., 13, Briggate, Leeds.  
Savage, W. W., 109, St. James's Street, Brighton.  
Savory, A. L., 143, New Bond Street, W.  
Sawyer, A. E., 100, High Street, Southwold.  
Sayer, E. C., 7, Warrington Road, Ipswich.  
Sayers, W. C., 63, High Street, Lewisham.  
Schaer, F., Badenia, Bridle Road, Purley.  
Schofield, Fred E., Newgate Street, Morpeth.  
Sclater, D. H., 17, Smith's Place, Edinburgh.  
Scruton, Saml. (Messrs. Raimes & Co.), Micklegate House, York.  
Selby, E. H., 104, Golden Lane, E.C.  
Self, P. A. W., B.Sc., F.I.C., 57, Chancery Lane, W.C.  
Sendall, C. E. J., Crosby Lea, Aintree, Liverpool.  
Senior, J., 2, Compton Street, Eastbourne.  
Seyler, Clarence A., B.Sc., F.I.C., Public Analyst, Nelson Terrace, Swansea.  
Seymour, F. S., The Square, Wimborne.  
Shacklady, J., F.C.I.S., Idlesse, Lyndhurst Road, Wallasey, Cheshire.  
Shacklock, J. H., 239, Streatham High Rd., S.W.  
Shadforth, W., 63, Grove Road, Bow, E.  
Shakespeare, W., Russell House, Walmley, near Birmingham.  
Sharp, Gordon, M.D., 9, Cavendish Road, Leeds.  
Sharvill, Frank, Hartfield, Staines, Middlesex.  
Shattock, John B., Prospect Street, Lancaster.  
Shaw, A., Riddings, Derbyshire.  
Shaw, A., 43, Green Lane, Stoneycroft, Liverpool.

- Shaw, J. W., 4, Edwardes Terrace, Kensington Road, W.  
 Shearer, J. A., 15, Beechgrove Avenue, Aberdeen.  
 Shears, James C., A.M.I.C.E., Farringdon Works, Shoe Lane, E.C.  
 Shelley, F. F., F.I.C., Apothecaries' Hall, Blackfriars, E.C.  
 Sheppard, W. F. J., F.C.S., 12, Bridge Street Row, Chester.  
 Shepherd, J. W., Settle, Yorks.  
 Sheppard, W. S., 54, Beak Street, Regent Street, W.  
 Shewell, A. B., 41, Parkhill Rd., Hampstead, N.W.  
 Shirtcliff, W. E. D., 66, Goldhawk Rd., Shepherds Bush, W.  
 Shorthouse, Herbert S., F.C.S., 144, Edmund Street, Birmingham.  
 Shuttlewood, W. B., F.C.S., c/o A. S. Watson & Co., 64, Crutched Friars, E.C.  
 Simmons, W. H., 96, Victoria St., Westminster, S.W.  
 Simon, J., 4, Eastgate Row, Chester.  
 Simpson, Thos., The Crofts, Hepscott, Morpeth.  
 Simpson, T. Munro, 24, Newgate Street, Newcastle-on-Tyne.  
 Skinner, H., The Dispensary, Great Northern Central Hospital, Holloway, N.  
 Smail, J. C., 8, North Methuen Street, Perth.  
 Small, J., Armstrong College Newcastle-on-Tyne.  
 Smallwood, F. W., 111, Grove Lane, Handsworth, Birmingham.  
 Smiley, John R., 111, Eccles Old Road, Manchester.  
 Smith, Arthur R., M.Sc., 3, Woodsley Terrace, Leeds.  
 Smith, Bernard, 6, Weymouth St., Portland Place, W.  
 Smith, E. H., 47, High Street, Gosport.  
 Smith, F., 221, Soho Rd., Handsworth, Birmingham.  
 Smith, J., 22, Chapel Rd., West Norwood, S.E.  
 Smith, J. B., c/o Messrs. Ransom & Son, Ltd., Hitchin.  
 Smith, J. L., 32, Ash Street, Southport.  
 Smith, John, 3, Terenure Road, Dublin.  
 Smith, J. Collett, 13, Cumberland Park, Acton, W.  
 Smith, Mrs. L. Wood, 9, Blenheim Road, Bedford Park, W.  
 Smith, Professor H. L., B.Sc., F.I.C., 17, Bloomsbury Square, W.C.  
 Smith, Roderick, 50, Point Street, Stornoway.  
 Smith, S., Ph.D., 2, Sunbeam Cottages, Dartford Heath, Kent.  
 Smith, T. Johnstone, 150, Holborn, E.C.  
 Smith, W. H., 77, Wennington Road, Southport.  
 Smithson, J., 1, Preston Road, Brighton.  
 Solomon, Albert H., 35, Mortlake Road, Kew Gardens, Surrey.  
 Somerton, W. K., 357, Battersea Park Rd., S.W.  
 Southall, A., F.C.S., Carrick House, Richmond Hill, Edgbaston, Birmingham.  
 Southall, A. Wm., Lower Priory, Birmingham.  
 Sparrow, A. B., 143, Highland Road, Southsea.  
 Speedie, Robert, Crieff, N.B.  
 Squire, P. W., F.L.S., F.C.S., 413, Oxford Street, W.  
 Stacey, H. G., F.L.S., F.C.S., 673, Commercial Road East, E.  
 Stainer, J. W., F.C.S., 71, Sandgate Road, Folkestone.  
 Stephens, H. I., 87, Barcombe Avenue, Streatham Hill, S.W.  
 Stephenson, Thos., F.C.S., F.R.S.E., 6, South Charlotte Street, Edinburgh.  
 Stevenson, H. Ernest, F.C.S., 122, Great Suffolk St., London, S.E.  
 Stevenson, W., 86, Palmerston Road, Southsea.  
 Steward, Alderman J. A., J.P., Fort Royal, Worcester.  
 Stewart, A. K., 1A, Lynedoch Place, Edinburgh.

Stiles, H. W., 30, Axholme Rd., Doncaster.  
 Stiles, M. H., F.R.M.S., 10, Avenue Road, Doncaster.  
 Stockman, Prof. R., M.D., F.R.C.P.E., The University, Glasgow.  
 Stocks, A. Booth, Chemist, Withington, Manchester.  
 Stones, Lionel, 60, Nassau Road, Barnes, S.W.  
 Stones, William, 7, Ardwick Green North, Manchester.  
 Stooke, F. A., 7, Station Parade, Sanderstead.  
 Storey W. A., Home Cottage, Wick, Bristol.  
 Storey, Mrs. W. A., A.I.C., Home Cottage, Wick, Bristol.  
 Storror, D., 228, High Street, Kirkcaldy, N.B.  
 Stout, Harry, 57, Broad Street, Pendleton, Manchester.  
 Strachan, A. L., 1, Alford Place, Aberdeen.  
 Strongitharm, W. G., 112, Upper George's Street, Kingstown,  
 Co. Dublin.  
 Sturton, J. G., 42, Bridge Street, Peterborough.  
 Surfleet, A. G., 221, Anlaby Rd., Hull.  
 Suttie, J. H. C., 9, Newkirk, Bearsden.  
 Swinton, T. H., 53, Oriel Road, Bootle.  
 Symes, C., Ph.D., F.C.S., 14, Hardman Street, Liverpool.

Tait, Joseph, 36, York Place, Edinburgh.  
 Talintyre, W. J., 193, Lake Lane, Liscard.  
 Tallantyre, S. B., B.Sc., F.I.C., 27, Fleet Street, Liverpool.  
 Tanner, A. E., F.C.S., Westminster Hospital, S.W.  
 Tawell, T. Edward, 9, Wellesley Road, Harrow.  
 Taylor, A. L., The Dispensary, Royal Infirmary, Bristol.  
 Taylor, C. Sansom, 224, Evering Road, Upper Clapton, N.E.  
 Taylor, F. W., 36, High Street, Newport Pagnell.  
 Taylor, J., 132, Irongate, Glasgow.  
 Taylor, Samuel, 3, Market Place, Derby.  
 Tharratt, G. R., 7, Myrtle Street, Liverpool.  
 Thomas, J. O., Royal South London Dispensary, London, S.E.  
 Thompson, A. W., Guy's Hospital, London, S.E.  
 Thompson, C., 159, Stratford Road, Sparkbrook, Birmingham.  
 Thompson, E., 8, Mayflower Road, Clapham, S.W.  
 Thompson, Edwin, Manestry Buildings, College Lane, Liver-  
 pool.  
 Thompson, H. A., 40, Aldersgate Street, E.C.  
 Thompson, J. A., 48, The Broadway, London Fields, N.E.  
 Thomson, W., F.I.C., F.R.S.E., Royal Institution Laboratory,  
 Manchester.  
 Thomson, W., 153, Byres Road, Glasgow.  
 Thornton, C. H., Beacon Hill, Exmouth.  
 Thorp, E. F. W., 96, Princess Road, Moss Side, Manchester.  
 Thorp, Walter, B.Sc. (Lond.), B.Sc. (Leeds), F.I.C., Dalkey, Co.  
 Dublin.  
 Tickle, T., B.Sc., F.I.C., Public Analyst's Laboratory, 83, Queen  
 St., Exeter.  
 Tirrell, J., The Square, Hanley.  
 Tocher, G. A., 329, High Holborn, W.C.  
 Tocher, J. F., D.Sc., F.I.C., 41½, Union Street, Aberdeen.  
 Tollitt, W., 111, Montague Street, Worthing.  
 Toplin, J. H., New Square, Chesterfield.  
 Towers, W. L., 10, Railway Street, Chatham.  
 Town, G. E., Evelina Hospital, Southwark Bridge Rd., S.E.

Truman, Frank W., 71, Old Kent Road, S.E.  
 Turner, C. W., 12, Foregate, Worcester.  
 Turner, J. Tyrie, Carrick-on-Suir, Ireland.  
 Turney, J. Davy, 183, Union Street, Plymouth.  
 Twining, Thos. C., 21, Cross Road, Chorlton-cum-Hardy, near Manchester.  
 Twivey, A., 151, Broad Street, Birmingham.  
 Tyrer, Thos., F.I.C., F.C.S., Stirling Chemical Works, Abbey Lane, Stratford, E.  
 Tyson, J., 16, Mayfield Road, Whalley Range, Manchester.  
 Umney, C., F.I.C., F.C.S., 48 and 50, Southwark Street, S.E.  
 Umney, E. A., 48 and 50, Southwark Street, S.E.  
 Umney, John C., F.C.S., 48 and 50, Southwark Street, S.E.  
 Uppill, W. T., 102, Rodney Rd., Walworth, S.E.

Vallance, A. C., Rowley Bank, Ellesmere Park, Eccles.

Walker, A., 26, Craighall Road, Trinity, Edinburgh.  
 Walker, F., Little Sutton, Cheshire.  
 Walker, John, 32, Virginia Street, Glasgow.  
 Wallis, T. E., B.Sc., F.I.C., F.C.S., 5, Hillsboro' Avenue, Exeter.  
 Walmsley, S. E., 8, Surbiton Park Terrace, Kingston-on-Thames.  
 Walsh, Dr. J. A., 30, Westmoreland Street, Dublin.  
 Walshaw, R. Carnelly, 4, Market Place, Huddersfield.  
 Want, W. Phillip, 194, Bishopsgate, E.C.  
 Ward, G., F.I.C., F.C.S., Millgarth Mills, Leeds.  
 Ward, J., 39, Eastgate Street, Gloucester.  
 Wardle, Miss E., Queen's Hospital, Bethnal Green, N.E.  
 Warner, C. Horne, B.Sc., F.I.C., 24, Gordon Street, Gordon Square, W.C.  
 Warrick, F. W., 6, Nile Street, City Road, N.  
 Watkins, A. Greenwood, Hainault, Laburnham Road, Maidenhead.  
 Watkinson, H. A., 149, Market Street, Farnworth, R.S.O.  
 Watson, A. J., 29, Front St., Tynemouth.  
 Watson, David M., 61, South Gt. George's Street, Dublin.  
 Watson, F. P., F.C.S., 6, Bailgate, Lincoln.  
 Watson, H. S., 223, Finchley Rd., London, N.W.  
 Watts, Eric, Balmoral House, Stalybridge.  
 Webb, E. A., 60, Bartholomew Close, E.C.  
 Webb, Harold E., 60, Bartholomew Close, London, E.C.  
 Webb, John H., Market Place, Luton.  
 Webb, Stephen F., 60, Bartholomew Close, London, E.C.  
 Weddell, George, 20, West Grainger Street, Newcastle-on-Tyne.  
 Weir, Alex. S., Kemnay, Aberdeenshire.  
 Wellcome, H. S., Snow Hill Buildings, Holborn Viaduct, E.C.  
 Wellings, Wm., 4, The Quadrant, Hoylake, Cheshire.  
 Wells, W. F., 20, Upper Baggot Street, Dublin.  
 Weston, J. H., Devonshire Buildings, Runcorn.  
 Whales, T., 41, London Rd., Southwark, S.E.  
 Whatmough, W. A., B.Sc., c/o Maw, Son & Sons, Aldersgate St., E.C.  
 Wherly, C., Raike Lane, Liscard.  
 White, Arthur F., 59 and 61, Sunbridge Road, Bradford, Yorks.  
 White, Edmund, B.Sc., F.I.C., 16, Cross St., Hatton Garden, E.C.

- White, J. F., Pendleton House, Spencer Place, Leeds.  
 White, Thos. A., Elm Grove, Southsea.  
 Whitehead, H. J., 67, Stanley Road, Bootle.  
 Whitfield, G., 113, Westborough, Scarborough.  
 Whitfield, J., F.C.S., 113, Westborough, Scarborough.  
 Whittle, Jas., F.C.S., 30, Bridge Street, Morpeth.  
 Whyte, J. S., 57, Guthrie Port, Arbroath, N.B.  
 Widdowson, T. S., 19, St. Margaret's Road, Brookley, S.E.  
 Wild, John, 307, Oxford Road, Manchester.  
 Wild, Sydney, 76, Mill Street, Macclesfield.  
 Will, Mrs. Watson, Mount Lodge, 9, Streatham Hill, S.W.  
 Willcox, W. H., M.D., F.R.C.P., B.Sc., 40, Welbeck Street, Cavendish Square, W.  
 Williams, D. J., 6, Cleveland Place East, Bath.  
 Williams, G. A., 131, Embden Street, Manchester.  
 Williams, Jesso, 132, Queen Street, Cardiff.  
 Williams, H. G., 118, The Moor, Sheffield.  
 Williams, Miss G. M., St. Paul's Eye Hospital, Liverpool.  
 Williams, T. R., Arnold Lodge, Church Road, Shortlands, Kent.  
 Williamson, Bamford, 4, Salisbury Place, South Shields.  
 Williamson, Ed., The Dispensary, Guy's Hospital, London, S.E.  
 Williamson, F. A., Moor Park Pharmacy, Garstang Road, Preston.  
 Williamson, H. S., 1, Camp Terrace, North Shields.  
 Williamson, Jas., 24, Heathside, Golders Green, N.W.  
 Williamson, L., 24, Newgate Street, Newcastle-on-Tyne.  
 Williamson, W. H., "Ashingdon," Wilmslow, Manchester.  
 Wilson, Harry, F.I.C., 32, Westwood Road, Southampton.  
 Wilson, J. F., 281, Essex Road, N.  
 Wilson, Thos., 110, High Street, Burntisland, N.B.  
 Wilson, Wm. Potter, 36, High Street, Haddington, N.B.  
 Windmill, W. H., Pharmacist, H.M. Prison, Wandsworth Common, S.W.  
 Wing, A. J., 69, Powis St., Woolwich.  
 Wokes, T. S., Grassendale, near Liverpool.  
 Wolfe, Ernest E., L.P.S.I., F.C.S., Kinsale, Co. Cork.  
 Wood, G. L., 21, Milton Road, Cambridge.  
 Wood, J., 158, Poulton Road, Seacombe.  
 Woodcock, R. C., F.I.C., F.C.S., Sanitas Co., Ltd., Locksley Street, Limehouse, E.  
 Woodhead, S. A., M.Sc., F.I.C., F.C.S., The County Laboratory, Uckfield, Sussex.  
 Woodruff, Thos., 43, Lapwing Lane, West Didsbury, Manchester.  
 Woods, W. H., 50, Bedford Street, Plymouth.  
 Woolcock, W. J. Uglow, Barrister-at-Law, 17, Bloomsbury Square, W.C.  
 Woolcombe, Dr. Robert Lloyd, M.A., LL.D. (Dublin Univ.), LL.D. (National Univ.), F.I.Inst., F.R.C.Inst., F.R.G.S., F.R.E.S., F.S.S., M.R.I.A., F.R.S.A. (Ireland), Barrister-at-Law, 14, Waterloo Road, Dublin.  
 Woolley, E. J., Victoria Bridge, Manchester.  
 Woolley, G. S., Victoria Bridge, Manchester.  
 Woolley, Hermann, Victoria Bridge, Manchester.  
 Woolley, Percy, Victoria Bridge, Manchester.  
 Woolley, S. W., 58, North Hill, Highgate, N.  
 Wootton, H., B.Sc., London College of Pharmacy, 361, Clapham Road, S.W.

Woulff, Hugo, 180, Philip Lane, Tottenham, N.  
Wride, F. B., Wholesale Chemist, Southampton.  
Wright, A., A.K.C., 22, Hardwick Road, Palmers Green, N.  
Wright, G. Victor, 18, Cadzow Place, Edinburgh.  
Wright, H. C., 48 and 50, Southwark Street, S.E.  
Wright, R., F.C.S., 11, Eagle Parade, Buxton.  
Wyatt, Harold, 223, Stanley Road, Bootle, Liverpool.  
Wyley, W. F., Wheatley Street, Coventry.  
Wyman, J. S., 58/59, Bunhill Row, E.C.

Yates, C. G., 9, Upper Hamilton Road, Brighton.  
Young, J. Rymer, F.C.S., 40, Sankey Street, Warrington.  
Young, R. F., Lindum House, New Barnet.

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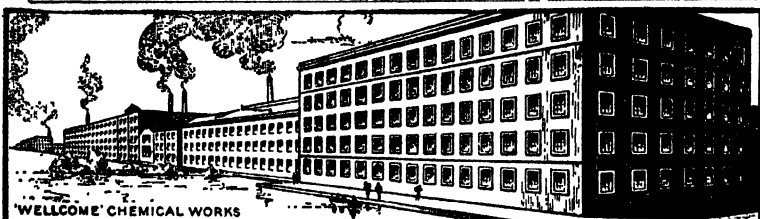






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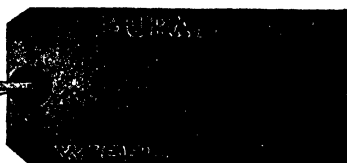
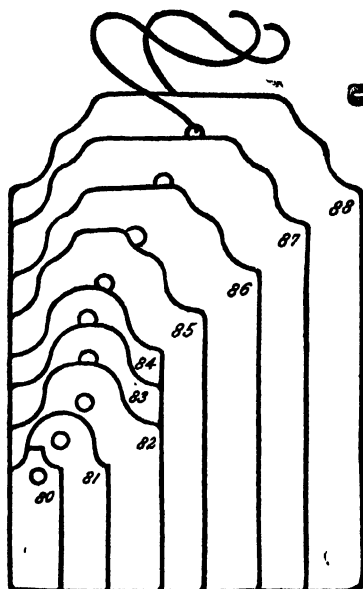
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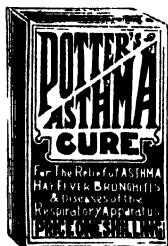
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